MULTIVESSEL BATCH DISTILLATION

SIGURD SKOGESTAD [1] , BERND WITTGENS, EVA SØRENSEN [2] and RAJAB LITTO

University of Trondheim, NTH,
Department of Chemical Engineering, 7034 Trondheim, Norway

Presented at: American Institute of Chemical Engineering, 1995 Annual Meeting,
November 12-17, 1995, Miami Beach, USA

Batch Process Modeling, Monitoring and Control, paper 184i

Abstract - The multivessel batch column presented in this paper provides a generalization of previously proposed batch distillation schemes. The economic potential of the multivessel batch distillation under total reflux is demonstrated by comparing with a conventional batch distillation column. A simple feedback control strategy for total reflux operation of a multivessel column is proposed. The feasibility of this strategy is demonstrated by simulations.

1 Introduction

Although batch distillation generally is less energy efficient than continuous distillation, it has received increased attention in the last few years because of its simplicity of operation, flexibility and lower capital cost. For many years academic research on batch distillation was focused primarily on optimizing the reflux policy. However, in most cases the difference to the simple-minded constant reflux policy usually is small. In practice, other issues are usually more important, such as the recycling of off-spec products, separation of azeotropic mixtures and pressure swing operation. More recently, one has started re-examining the operation of batch distillation as a whole. For example, for mixtures with a small amount of light component, a cyclic operation where the operation is switched between total reflux operation and dumping the product (i.e., the condenser holdup is introduced as an additional degree of freedom) may be better (Sørensen and Skogestad, 1994). The simplest operation strategy is with only one cycle, that is, the column is operated under total reflux and the final products are collected in the condenser drum and in the reboiler. Another alternative for this type of mixture is to "invert" the column by charging the feed to the top and removing the heavy product in the bottom (Robbison and Gilliland, 1950) . It has also been suggested to use a middle vessel where the feed is charged to the middle of the column (Bortolini and Guarise, 1970).

All these policies may be realized in a *multivessel batch distillation column* with both holdups and product flows as degrees of freedom. With N_c vessels along the column and with given pressure and heat input, this column has $2N_c - 1$ degrees of freedom for optimization; namely the $N_c - 1$ holdups (e.g., controlled by the $N_c - 1$ reflux streams) and the N_c product rates. A simplified flowsheet of the multivessel batch distillation column is shown in Figure 1.

The simplest operation form of the proposed multivessel column, which is the focus of this paper, is the *total reflux operation* where the N_c product rates are set to zero ($D_i = 0$). The total reflux operation of a conventional batch distillation without a middle vessel was suggested independently by Bortolini and Guarise (1971) and Treybal (1970). Hasebe et al. (1995) proposed a process based on total reflux operation where one can separate more than two components, they denote this process a "multi-effect batch distillation system". There are at least two advantages with this multivessel column compared to conventional batch distillation where the products are taken over the top, one at a time. First, the operation is simpler since no product change-overs are required during operation. Second, the energy requirement

[1] Author to whom correspondence should be addressed, fax: +47 7359 4080; E-mail: skoge@kjemi.unit.no
[2] Centre of Process System Engineering, Imperial College, London, SW7 2 AZ

may be much less due to the multi-effect nature of the operation, where the heat required for the separation is supplied only to the reboiler and cooling is done only at the top. In fact, Hasebe et al. (1995) show that for some separations with many components the energy requirement may be similar to that for continuous distillation using $N_c - 1$ columns.

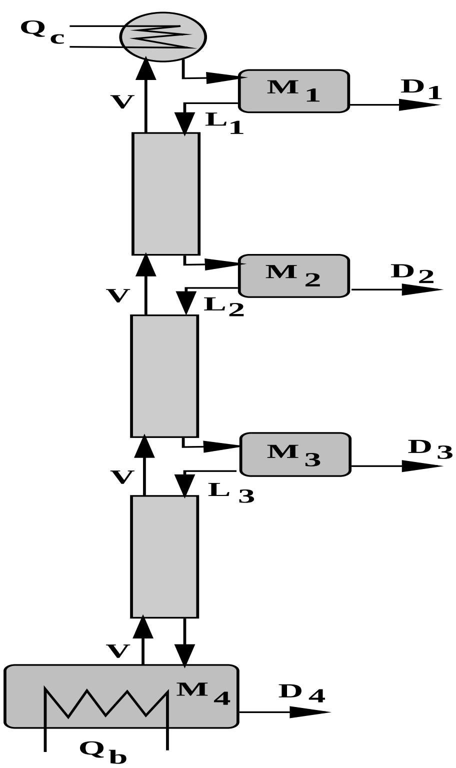

Figure 1: General multivessel batch distillation column

Hasebe et al. (1995) propose to "control" the total reflux multivessel batch distillation column by calculating in advance the final holdup in each vessel and then using a level control system to keep the holdup in each vessel constant. For cases where the feed composition is not known exactly they propose to, after a certain time, adjust the holdup in each vessel based on composition measurements. Their scheme, involving the optimization of the vessel holdups and their adjustment based on composition measurement in these vessels, is rather complicated to implement and requires an advanced control structure to implement the control law.

We propose a feedback control structure based on $N_c - 1$ temperature controllers (see Fig. 3). The idea is to adjust the reflux flow out of each of the upper $N_c - 1$ vessels by controlling the temperature at some location in the column section below. There is no explicit level control, rather the holdup in each vessel is adjusted indirectly by varying the reflux flow to meet the temperature specifications.

The paper is divided into 6 parts. First we present the principle of operation and a simulation example to show the feasibilty of the proposed process. Conventional and multivessel batch distillation are compared on a qualitative basis and the economical potential of the new process is shown. The dynamic models are implemented in the SPEEDUP environment (Speedup, 1993). In the fourth part we present the proposed implementation of the multivessel column and dynamic simulation results of its operation. Finally the results are compared and conclusions are presented.

2 Total Reflux Operation with constant vessel holdups

In this section we follow Hasebe et al. (1995) and present simulations which demonstrate the feasibility of the multivessel batch distillation under total reflux. The holdup of each vessel is calculated in advance by taking into account the amount of feed, feed composition and product specifications. After feeding the predescribed amount of raw material to the vessels, total reflux operation with constant vessel holdup is carried out until the compositions in all vessels satisfy their specifications.

Table 1: Summary of column data and initial conditions

Number of components	$N_c = 4$
Relative volatility	$\alpha_i = [10.2, 4.5, 2.3, 1]$
Total number of stages	$N_{tot} = 33$
Number of sections	$N_s = 3$
Number of stages per section	$N_t = 11$
Vessel holdup	$M_m = 2.5\ kmol$
Tray holdup	$M_t = 0.01\ kmol$
Total initial charge	$M_{tot} = 10.33\ kmol$
Reflux flow	$L = 10\ kmol/hr$
Vapor flow	$V = 10\ kmol/hr$

The numerical value of ratios of the relative volatilities is chosen to be close to the experimental system in the pilot plant. Constant molar flows are assumed.

Typical simulated composition profiles as a funtion of time are shown in Figure 2 for a 4-component mixture with an initial feed composition of

$$z_{F,1} = [0.25, 0.25, 0.25, 0.25] \quad (1)$$

Data for the column and feed mixture are given in Table 1.

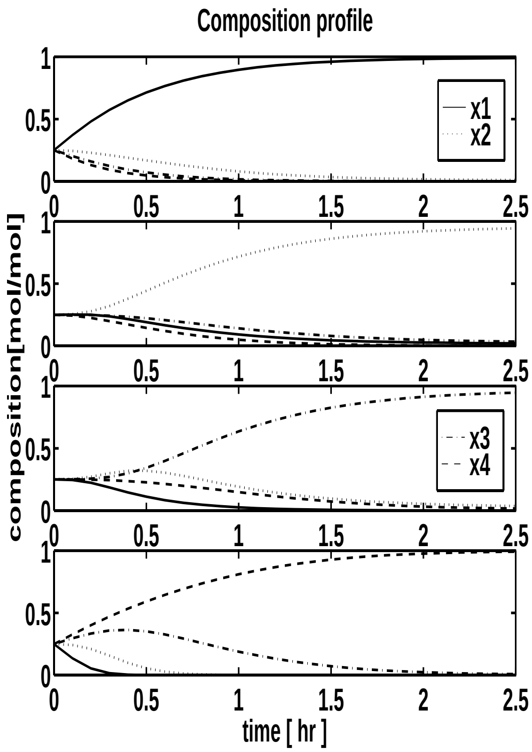

Figure 2: Constant vessel holdup: Composition response in accumulator, vessel 2, vessel 3 and reboiler (from top) for feed mixture $z_{F,1}$

As time goes to infinity the steady state compositions presented in Table 2 are achieved. However, the operation policy of keeping the holdup of the vessels constant may be difficult to achieve in practice and also is very sensitive to errors in the assumed feed compositon.

Table 2: Steady state composition for initial feed composition $z_{F,1}$; Constant vessel holdups $M_i[kmol]$

	Vessel 1	Vessel 2	Vessel 3	Vessel 4
M_i	2.5	2.5	2.5	2.5
x_1	0.993	0.017	0.0	0.0
x_2	0.007	0.959	0.025	0.0
x_3	0.0	0.024	0.963	0.004
x_4	0.0	0.0	0.012	0.996

The last problem is illustrated by considering a case where the actual feed composition is

$$z_{F,2} \;=\; [0.30, 0.10, 0.40, 0.20] \quad (2)$$

but the holdup of each vessel is equal to the example with feed composition $z_{F,1}$ in Eq. 1. This results in large changes in the final vessel compositions as seen from Table 3. For example, the purity in vessel 2 is reduced from $x_2 = 0.959$ to $x_2 = 0.404$.

Table 3: Steady state composition for initial feed composition $z_{F,2}$; Constant vessel holdups $M_i[kmol]$

	Vessel 1	Vessel 2	Vessel 3	Vessel 4
M_i	2.5	2.5	2.5	2.5
x_1	0.999	0.203	0.0	0.0
x_2	0.001	0.404	0.001	0.0
x_3	0.0	0.393	0.999	0.180
x_4	0.0	0.0	0.0	0.820

To compensate for these feed variations Hasebe et al. (1995) propose a rather complicated algorithm for adjusting the holdup based on measuring the composition in the vessels. We propose a much simpler scheme which is discussed in section 4.

3 Economic potential of the Multivessel Batch column

Hasebe et al. (1995) found that the energy efficiency of the multivessel batch distillation could be compareable to continuous distillation. We compare the energy usage in sense of the final batch time (t_f) with conventional batch distillation for the initial feed composition $z_{F,1}$ and a vapor flow of $V = 10\ kmol/hr$. The number of stages for the conventional batch column is equal to the sum of stages for the multivessel batch column. A constant reflux policy is used for the conventional column. The product specification $x_{spec} = [0.99, 0.95, 0.95, 0.99]$ is in both cases, given as a lower bound on the purity of the main component in all four products.

The amount and compositions of products obtained by conventional and multivessel batch distillation are presented in Tables 4 and 5, respectively. We find that the conventional batch column requires at least

70 % more energy ($L_1/V = 0.85$) and produces approximately 10 % off-cut until all four product specifications are fulfilled. A minimum amount of offcut is achieved with a reflux ratio of $L_1/V = 0.9$, this requires approximately 150 % more energy than the multivessel batch column. For reflux ratios $L_1/V \leq 0.8$ the required product specifications are not fulfilled.

Table 4: Comparison of holdup, off-cut amount and final batch time for multivessel and conventional batch distillation

	L_1/V	Product 1	Product 2	Product 3	Product 4	Off-cut	$t_f[hr]$
Multivessel	1	2.506	2.452	2.512	2.530	0.0	3.39
Conventional	0.80	2.259	1.703	2.103	1.853	2.081	4.57
	0.85	2.415	2.317	2.441	2.091	0.736	5.77
	0.9	2.523	2.619	2.537	2.321	0.0	8.18

Table 5: Comparison of the achieved product quality of multivessel and conventional batch distillation

	L_1/V	Product 1	Product 2	Product 3	Product 4
Multivessel	1	0.993	0.967	0.960	0.993
Conventional	0.80	0.990	0.949	0.938	0.990
	0.85	0.990	0.950	0.947	0.990
	0.9	0.990	0.950	0.972	0.989

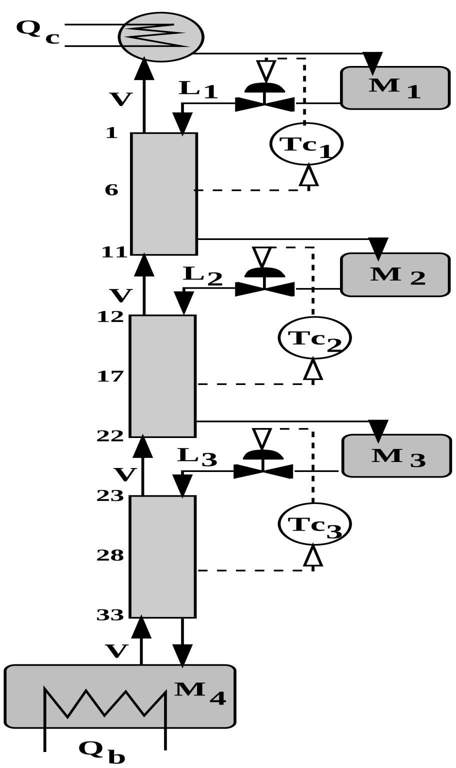

Figure 3: New feedback control structure for multivessel batch distillation column under total reflux

4 A NEW FEEDBACK CONTROL STRUCTURE

A flowsheet of our proposed control structure is shown in Figure 3. The separation of a mixture containing N_c components require N_c vessels and $N_c - 1$ temperature controllers. The temperature controller (Tc_2) adjust the reflux flow (L_2) out of the vessel (M_2) above that column section. This enables an indirect control of the holdups in the vessels. Note that there is no level controller or level measurement, although some minimum and maximum level sensors may be needed for safety reasons.

The setpoints for each temperature controller may be, in the simplest case, set as the average boiling temperature of the two components being separated in that column section, these setpoints are used in the simulations presented below. Alternatively, they may be obtained by steady-state calculations to get a desired separation, or they may be optimized as a function of time.

To demonstrate the feasibility of our proposed control scheme we consider the same example as studied above (see Table 1). The utilized controllers are simple proportional controllers as given in table 6.

The proportional control algorithm is:

$$L_i = K_c \cdot (T_i - T_{s,i}) \tag{3}$$

For simplicity the column temperature is assumed to be the average of the boiling temperatures

$$T = \sum_{i=1}^{N_c} x_i \cdot T_{b,i} \tag{4}$$

where $T_{b,i} = [64.7, 78.3, 97.2, 117.7]^oC$.

Table 6: Data for temperature controllers

	$T_{s,i}$	K_c	location *
	^oC	$^oC/kmol$	
Tc_1	71.5	-0.25	6
Tc_2	87.75	-0.25	17
Tc_3	107.2	-0.25	28

* *counting top to bottom*

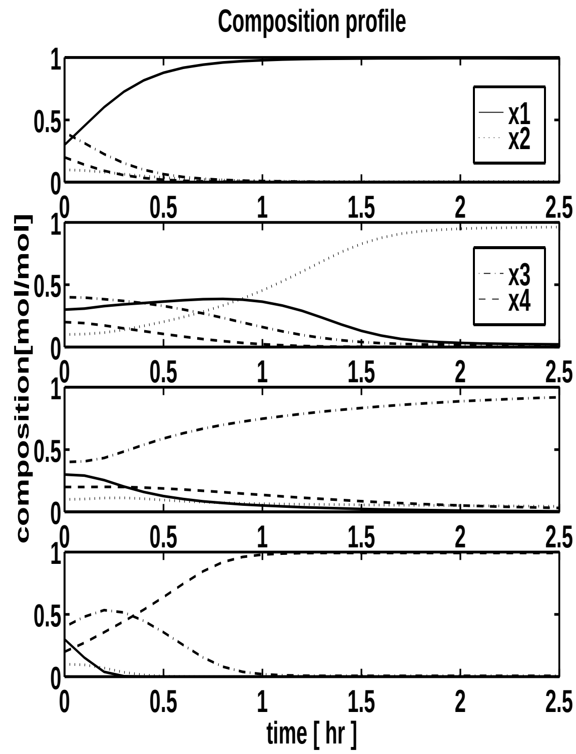

Figure 4: Temperature control: Composition response in vessels 1, 2, 3, and 4 for feed mixture $z_{F,2}$

Table 7: Steady state compositions obtained by distillation with feedback control

	Vessel 1	Vessel 2	Vessel 3	Vessel 4
x_1	0.993	0.016	0.0	0.0
x_2	0.007	0.967	0.034	0.0
x_3	0.0	0.017	0.960	0.007
x_4	0.0	0.0	0.006	0.993

To demonstrate that the proposed control scheme is insensitive to the initial feed composition we use two different initial feed compositions, $z_{F,1}$ (Eq. 1) and $z_{F,2}$ (Eq. 2). In *both* cases the same steady state compositions (see Table 7) are reached as $t \rightarrow \infty$. However the resulting holdups in the vessels is different in each case, as shown in Table 8.

Table 8: Steady state holdup distribution for feed compositions $z_{F,i}$

	Vessel 1	Vessel 2	Vessel 3	Vessel 4
feed	M_1	M_2	M_3	M_4
	$[kmol]$	$[kmol]$	$[kmol]$	$[kmol]$
$z_{F,1}$	2.506	2.452	2.512	2.530
$z_{F,2}$	3.053	0.788	4.159	2.000

The composition profiles as a function of time are not shown for mixture $z_{F,1}$ since these are quite similar to these for the case with constant holdup as shown in figure 2. (The approach to equilibrium is somewhat faster in vessel 1 and 4 and slower in vessel 2 and 3.) On the other hand with feed composition $z_{F,2}$ the new policy ensures that the required product qualities are achieved. This is seen from Tables 7 and 8 and is further demonstrated in Figures 4 and 5, which show the composition profiles in the vessels (Fig. 4) and the holdup in the vessels (Fig. 5, top), flows out of the vessels (Fig. 5, center), and controlled temperatures (Fig. 5, bottom).

In Fig. 5 it is observed that the controlled temperatures reach their setpoint $T \rightarrow T_s$ as $t \rightarrow \infty$, even though only proportional controllers are used. The reason why we get no offset is that the model contains an integrator, since the system is closed. More specifically, consider the reflux L to a column section and the temperature T in that section. We know that we can change the steady state value of T by changing L. We also know that a steady state change in L is not allowed, since we have to have $L = V$ as $t \rightarrow \infty$ (total reflux operation). Thus the transfer function from L to T must contain an integrator.

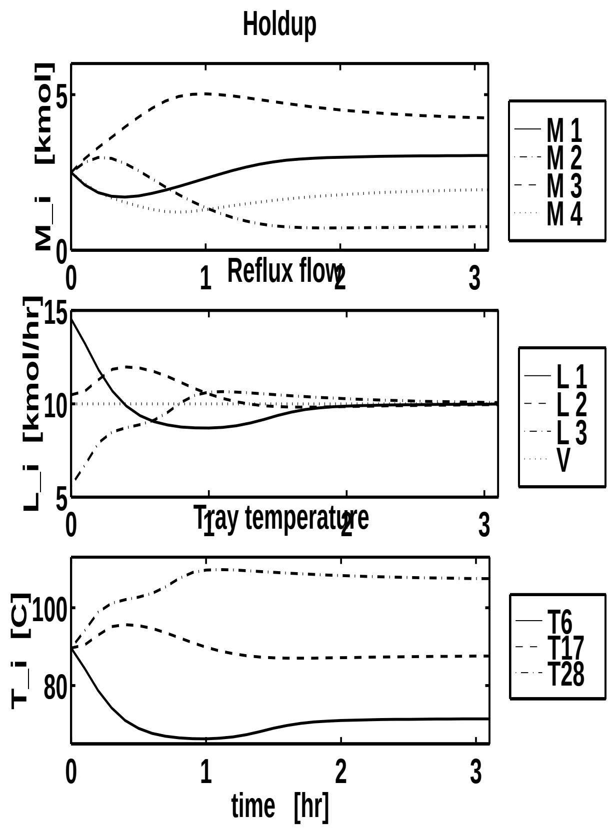

Figure 5: Temperature control: Responses for holdup, reflux flow and tray temperature for feed mixture $z_{F,2}$

With temperature control we achieve the same steady state compositions in the vessels independent of the initial feed composition (only vessel holdups differ at steady state). The reason is that the column has only three degrees of freedom at steady state and if we fix three temperatures at three locations in the column, then the temperature profile over the column at total reflux is determined (if we assume that we do not have multiple steady states). Multiple solutions are not likely when temperatures are specified, but may be encountered if we specify the composition of a given component.

We have also performed some simulations to study the start-up for the case when the entire feed mixture is charged to the reboiler (and not distributed to the vessels). The results indicate that the temperature controllers can be activated immediately after start-up or possibly after a short period with total reflux. The vessels are then slowly filled up by action of the temperature controllers which reduce the reflux flows for a transient period. The simulations indicate that one should exercise some care to avoid emptying the reboiler.

5 Discussion

Simulations for the case with $z_{F,1}$ presented in this paper indicate energy savings of approximately 50 %. In conventional batch distillation the optimal operation depends quite strongly on the reflux policy and the use of off-cuts to achieve the desired product composition. Optimizing the reflux policy and off-cut amounts for the desired separation enables the utilzation of one process for quite different separation problems. The implementation of the reflux policy (especially for more complicated profiles) and the switching between product and off-cut fractions require often an advanced control system or an experienced operator. On the other hand, in multivessel batch distillation there are fewer degrees of freedom and this simplifies the operation considerably. The reflux flow is controlled by simple proportional controllers such that the desired products are accumulated in the vessels. One disadvantage with the multivessel column compared with the conventional batch distillation is that the column itself is more complicated. Also, whereas in a conventional batch column one only has to make decision on the length of one single column section, one has to decide on the number of sections and their length for a multivessel column. The design of the multivesel columns is therefore more closely linked to a specific feed mixture, relative volatility and product specifications. Thus, the design process of a multivessel column is similar to the design of a sequence of continuous distillation columns.

Although the work presented here is rather encouraging from a viewpoint of practical implementation many questions remain before this process will be accepted by industry. Further work will include:

1. Perform experiments on a newly build pilot plant to verify the simulation results.
2. Study improved methods for practical operation. *e.g.:* controller types and start-up procedures.
3. Study the effect of optimizing the temperature setpoints over time and implementation of the setpoint trajectories.
4. Determine the types of mixtures and conditions which are most suited for the separation in the new process.

5. Determine the optimal initial liquid distribution over the column.

6 Conclusions

A new control strategy for the multivessel batch distillation column is proposed. It is shown that the proposed control scheme is easy to implement and operate, even for widely varying feed compositions. By simulation it is found that the multivessel batch distillation column is more energy efficient than a conventional batch distillation column where the products are withdrawn over the top.

Notation

D	Distillate flow rate $[kmol/hr]$
K	Controller gain
L	Reflux flow rate $[kmol/hr]$
M	Molar holdup $[kmol]$
M_{tot}	Total inital feed charge $[kmol]$
N_c	Number of components
N_t	Number of stages per section
N_{tot}	Total number of stages
Q_b	Reboiler heat duty
Q_c	Condenser heat duty
R	Reflux ratio
T	Temperature
t	time
V	Molar vapor flow
x	Liquid composition
y	Vapor composition
z	Feed composition
α	Relative volatilty

Subscripts

b	Boiling point
F	Feed
f	final
i, j, k, l, m	Identifier
off	Off-cut
R	Reboiler
s	Controller Setpoint
t	Total number
tot	Total amount

References

[1] Bortolini, P. and Guarise, G.B., “Un nuovo metodo di distillazione discontinua; (A new method of Batch Distillation)”, Ing. Chim. Ital., Vol. 6, No. 9, Sep. 1970

[2] Hasebe, S., B. Abdul Aziz, I. Hashimoto and T. Watanabe,”Optimal design and operation of complex batch distillation Column”, *Preprint of the IFAC Workshop on Interaction between Process Design and Process Control*, London,

[3] Hasebe, S., T. Kurooka and I. Hashimoto, ”Comparison of the separation performances of a multi-effect batch distillation system and a continuous distillation system”, *Proc. IFAC-symposium DYCORD'95*, Denmark, June 1995

[4] Robinson, C.S. and E.R. Gilliland, “Elements of Fractional Distillation”, *McGraw Hill Book Company.*, New York, 4th ed., 1950

[5] SPEEDUP Release 5.4 User Manual, Prosys Technology Ltd., 1993

[6] Sørensen, E. and S. Skogestad, “Optimal operating policies of batch distillation”, *Proc. Symposium PSE'94*, Kyongju, Korea, June 1994, 449-456

[7] Treybal, R.E., ”A Simple Method of Batch Distillation”, *Chemical Engineering*, Oct. 1970

Appendix

Mathematical Model of Multibatch Distillation

The mathematical model for the multivessel batch distillation column is based on the model of a conventional batch distillation column.

The proposed model is based on the following assumptions:

- constant relative volatility
- constant molar liquid and vapor flow (neglect flow dynamics)
- energy balance neglected
- constant pressure
- constant molar holdup on the stage
- constant tray efficiency (100 %)
- negligible vapor holdup
- perfect mixing on all trays and in all vessels
- total condenser

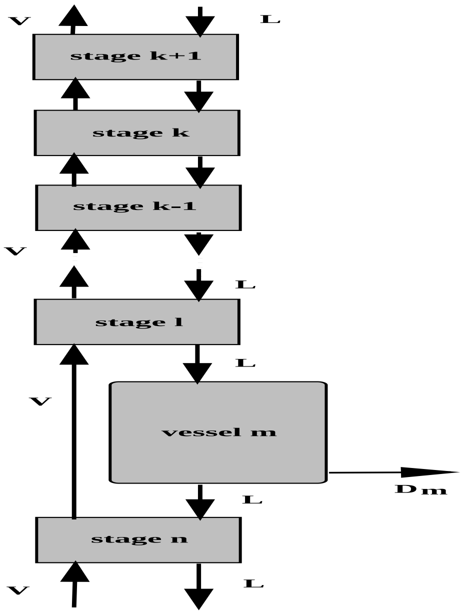

Figure 6: Connection of trays and vessels

The distillation column is modeled as a stack of stages (counted from top). The model for stage k consists of a mass balance for constant stage holdup

$$M_k \frac{dx_k}{dt} = L \cdot (x_{k+1} - x_k) + V \cdot (y_{k-1} - y_k) \quad (5)$$

and the vapor liquid equilibrium

$$\alpha_{i,j} = \frac{y_i/x_i}{y_j/x_j} \quad (6)$$

The modeling of the intermediate vessels and the condenser (note we assume a total condenser) follow the assumptions given previously. The mass balance is given by

$$\frac{d(M_l \cdot x_l)}{dt} = L \cdot (x_{l+1} - x_l) \quad (7)$$

Note that the vapor flow does not pass through the vessels. The reboiler is modeled by the following equation

$$\frac{d(M_R \cdot x_R)}{dt} = L \cdot x_{N_{tot}} - V \cdot y_R \quad (8)$$

where the vapor liquid equilibrium is described by equation 6. The mathematical description of the reboiler simplified by assuming a constant molar boilup rate.