# MULTIVESSEL BATCH DISTILLATION - EXPERIMENTAL VERIFICATION

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The experimental verification of the operation of a multivessel batch distillation column, operated under total reflux is presented. This column configuration, consisting of several column sections and intermediate vessels, provides a generalization of previously proposed batch distillation schemes. We propose a simple feedback control strategy which is capable to achieve a desired product composition in all product vessels, even for widely varying feed compositions. The idea is to use the reflux flow out of each vessel to control the temperature at some location in the section below. The practicality of the proposed control scheme has previously been demonstrated by dynamic simulations, and in this paper we present experimental results from a laboratory scale apparatus which verify these simulations.

Keywords: multivessel batch distillation, temperature control

## INTRODUCTION

As an alternative to conventional batch distillation, also called the batch rectifier, Robinson and Gilliland (1950) proposed an inverted batch column, also called the batch stripper, where the feed is charged to the top and the heavy products are drawn from the bottom of the column. A total reflux operation strategy, where the two final products are collected in the condenser drum and in the reboiler, was suggested by Treybal (1970).

An extension of batch distillation is the middle vessel column, which has both a rectifying and stripping section. This configuration was first mentioned by Robinson and Gilliland (1950) for separation of a binary mixture. Hasebe *et al.* (1992) proposed to charge a *ternary* mixture to the middle vessel, and let the light and heavy impurities be drawn from the top and the bottom of the column. In this case the operation stops when the intermediate component in the middle vessel has reached its desired purity.

A further generalization is to use several middle vessels as suggested by Hasebe *et al.* (1995). They proposed a total reflux operation where the products are collected in vessels along the column. Because one may view this column as a stacking of several columns on top of each other, they denote this process a "multi-effect batch distillation system" (MEBAD).

All the above designs and strategies can be realized in the *multivessel batch distillation column* shown in Figure 1, where the holdups, $M_i(t)$, are degrees of freedom.

The simplest strategy for operating the multi-vessel column, which is the focus of this paper, is the *total reflux operation* suggested by Hasebe *et al.* (1995). In this case, $N_c$ vessels are required to separate $N_c$ components into pure products. There are at least two advantages with the multivessel column compared to conventional batch distillation where

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the products are drawn 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 may be less due to the multi-effect nature of the operation. In fact, Hasebe *et al.* (1995) found that for some separations with many components the energy requirement may be similar to that for continuous distillation using $N_c - 1$ columns.

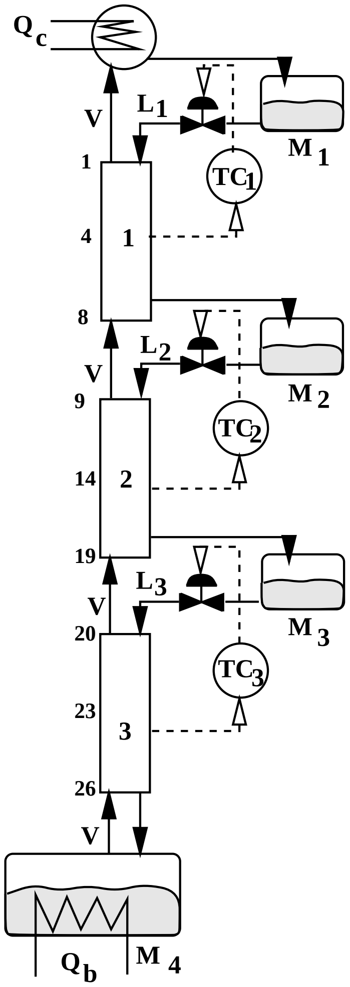


Figure 1: Feedback control structure for multivessel batch distillation column under total reflux. Stage numbers are given on figure.

The disadvantage with the multivessel column is that the design is more complicated. Also, the procedure for operation and control of product compositions suggested by Hasebe *et.al.* (1995), based on precomputing of the desired final holdup in each vessel, was rather complicated and sensitive to errors.

Instead, Skogestad *et.al.* (1995,1997) proposed a simple feedback control scheme for the operation of the multivessel column. The control structure is based on $N_c - 1$ temperature controllers (see Figure 1). 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, $M_i$, in each vessel is adjusted indirectly by varying the reflux flow to meet the temperature specifications. Their simulation study showed that the operation is feasible and that the final product compositions are independent of the initial feed composition.

The main contribution of this paper is to present the experimental verification of the proposed feedback control structure for the total reflux operation of the multivessel batch distillation column.

## INTRODUCTORY SIMULATION EXAMPLE

In this section, we present rather briefly, simulation results for the operation of the multivessel column applying a simple feedback control scheme. For further details, we refer to the paper by Skogestad *et.al.* (1997). A summary of column data and initial conditions is given in Table 1.

The relative volatilities in the simulation example are chosen to match the mixture used in the experiments, which consists of methanol, ethanol, n-propanol and n-butanol. For simplicity the column temperature is assumed to be the average of the boiling temperatures $T = \sum_{i=1}^{N_c} x_i \cdot T_{b,i}$ with $T_{b,i} = [64.7, 78.3, 97.2, 117.7]^o C$. The setpoints for each temperature controller may, in the simplest case, be set as the average boiling temperature of the two components being separated in that column section, $T_{s,i} = [71.5, 87.75, 107.2]$, and these values are used in the simulations below.

Table 1: Summary of column data and initial conditions

| | | | |
|---|---|---|---|
| Number of components | $N_c = 4$ | Relative volatility | $\alpha_j = [10.2, 4.5, 2.3, 1]^1$ |
| Total number of stages | $N_{tot} = 26^2$ | Number of sections | $3^3$ |
| Vessel holdups | $M_{i,0} = 2.5\ kmol$ | Tray holdups (constant) | $M_k = 0.01\ kmol$ |
| Reflux flows | $L_{i,0} = 10\ kmol/h$ | Vapor flow (constant) | $V = 10\ kmol/h$ |
| Total initial charge | $M_{tot} = 10.33\ kmol$ | | |

[1] *Adjusted to match mixture used in experiment;* [2] *Determined from experimental data (rounded to the next integer) excluding reboiler;* [3] *The distribution of trays over the sections is shown in Figure 1*

We use simple proportional temperature controllers to manipulate the reflux flow $L_i = K_c \cdot (T_i - T_{s,i}) + L_{i,0}$ with a controller bias of $L_{i,0} = V = 10\ kmol/h$. The numerical value of the controller gain is $K_c = -0.25 \frac{kmol}{h\ ^\circ C}$. The temperature sensors are located in the middle of each column section (trays 5, 14 and 23 from the top).

We here assume that the initial condition at start-up is to have all the feed charge as boiling liquid in the reboiler[2]. Vapor flow exiting from the reboiler is condensed in the top and recycled to the column after passing through the accumulator. The action of the reflux flow controllers, $TC_i$, will indirectly adjust the level in the intermediate vessels by temporary changing the molar reflux flow based on the temperature readings in the column.

Table 2: Steady-state holdup and compositions (simulations)

| | Vessel 1 | Vessel 2 | Vessel 3 | Vessel 4 |
|---|---|---|---|---|
| $M$ | 2.98 | 1.72 | 1.88 | 3.42 |
| $x_1$ | **0.964** | 0.036 | 0.0 | 0.0 |
| $x_2$ | 0.036 | **0.947** | 0.033 | 0.0 |
| $x_3$ | 0.0 | 0.017 | **0.935** | 0.036 |
| $x_4$ | 0.0 | 0.0 | 0.032 | **0.964** |

With the temperature setpoints given, we achieve for a feed charge of $M_{init} = 10.33\ kmol$ and a composition of $z_F = [0.27, 0.19, 0.20, 0.34]$ the steady-state liquid holdup and compositions ($t \to \infty$) given in Table 2. In fact, as shown by Skogestad *et.al.* (1996) the steady-state product compositions are independent of the intial feed composition.

In Figure 2 (a) and (b), we present composition time responses of main component and impurities in the vessels. The simulations demonstrate how the action of the temperature controllers adjust the reflux flows (see Figure 2 d), which indirectly adjust the vessel holdups (see Figure 2 c) such that the final products are of high purity. The results indicate that the temperature controllers can be activated immediately after start-up; possibly with some strategy to ensure that the vessels are not emptied. The vessels are then slowly filled up by action of the temperature controllers which reduce the reflux flows for a transient period. In Figure 2 (e) we present the controlled temperatures in the center of the column sections.

[2] Skogestad *et.al.* (1997) investigated two alternative start-up procedures for the multivessel batch distillation column

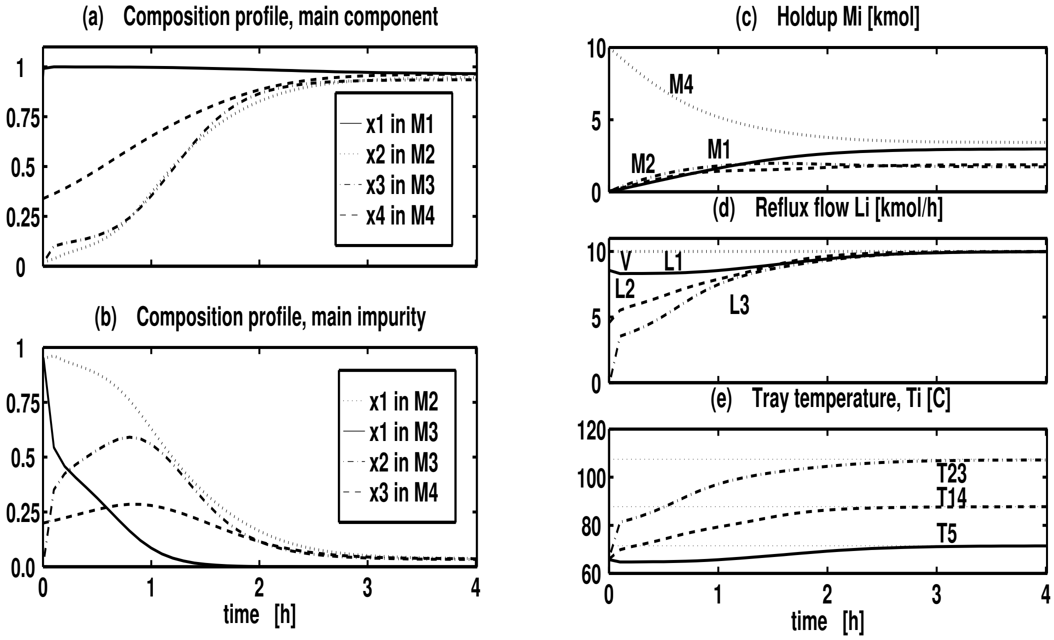


Figure 2: Simulations using temperature control: Vessel compositions (a), impurities (b), holdups (c), reflux flows (d) and tray temperatures (e) as a function of time

## MULTIVESSEL BATCH DISTILLATION PILOT PLANT

A laboratory scale multivessel batch distillation unit (see Figure 3) was build to perform the experiments needed to verify pervious presented simulation results. A summary of data of the experimental facility is given in Table 3.

The unit is built in glass and insulated to reduce heat loss to the surroundings during operation. Inspection openings facilitate monitoring of the liquid distribution over the system. The apparatus is operated at atmospheric pressure.

The multivessel batch distillation column consists of a 4 l reboiler; three intermediate vessels of 1 l volume and a total condenser. The liquid volume accommodated in the column during operation is approximately 4 liters, this holdup can be stored either in the reboiler or will be evenly distributed over all four vessels. The four vessels are connected by three packed column sections of 420 mm length and 30 mm diameter which are filled with double-wound wire mesh rings of 3 x 3 mm made from stainless steel by Normschliff.

The estimation of the number of theoretical stages of the laboratory column is based on binary vapor-liquid equilibrium data (Gmehling and Onken, 1977) and from the experimentally determined product compositions. The amount of lighter and heavier impurities are combined to a pseudo-component and a McCabe-Thiele diagram is then used to estimate the number of stages.

Each column section is equipped with three chromel-alumel-thermocouples placed in the center of the column cross section. Thermocouples are placed 5 cm from each end and a third in the middle of the column section. The thermocouples placed in the middle of the columns control the reflux from the vessels above by means of two-way solenoid valves which are operated by solid-state relays.

The setpoint of the reflux controller is set to the arithmetic middle of the boiling points of components which are expected to accumulate in two adjacent vessels. The chosen controller type for the experiments is a standard PI-controller which is tuned to be rather slow ($K_c = 1.3 \frac{ml}{^oC\ min}$, $\tau_I = 10\ min$) to avoid extensive control action during start-up and in presence of disturbances. The reflux is introduced to the center of the column, slightly above the packing material.

Thermocouples are also placed in the intermediate vessels and in the reboiler to give an indication of product qualities by measuring the temperature in the liquid phases. A second thermocouple installed in the reboiler measures the surface temperature of the heating element, thus by controlling the temperature difference between reboiler holdup and heating mantle the reboiler duty is adjusted.

Table 3: Data for the laboratory column

| | |
|---|---|
| Vessel holdup | $1 \leq M_m \leq 20\ mol$ |
| Tray holdup | $M_t \simeq 0.025\ mol$ |
| Total initial charge | $35 \leq M_{tot} \leq 45\ mol$ |
| Heat input reboiler | $Q_b \simeq 375\ W$ |
| Flows | $L \simeq V \simeq 32\ mol/h$ * |
| Boilup ratio | $M_{tot}/V \simeq 1.25\ h$ |

* *Note: The steady state vapor flow is estimated from* $V = Q_b/\Delta h_{vap}$

The process is interfaced to a PC-based control system from INTEC Controls Cooperation (Paragon FS 502, Ver. 2.32). All sensors installed at the plant are connected to a 12-bit data acquisition board, which operate at a sampling frequency of 1 Hertz. Product composition analysis is performed by means of a gas chromatograph. The chemical system studied is methanol, ethanol, n-propanol and n-butanol. This mixture is chosen due to the relative high relative volatility ($\alpha_{i,j} \geq 1.8$) and the fairly ideal behavior.

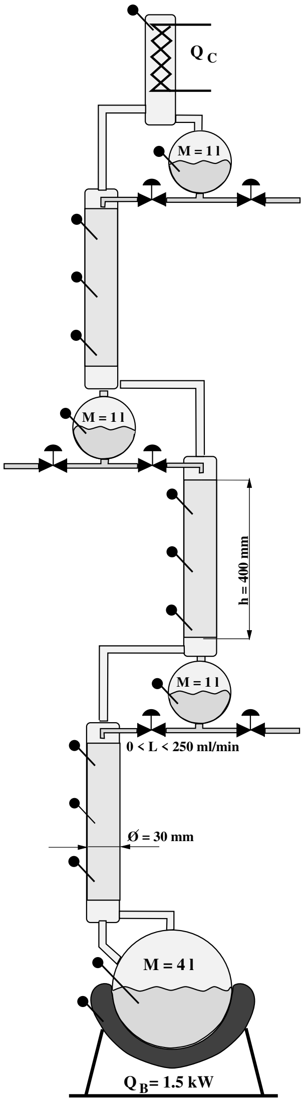


Figure 3: Pilot plant scale distillation column

## EXPERIMENTAL RESULTS

In Table 4 we present data from 8 experiments performed, where widely varying feed compositions are used. The results show, as expected from the introductory simulation example, that the final product composition (after about 6 hours of operation), $x_i(M_i)$, is independent of the initial feed composition $z_F$ for the new feedback control structure. A comparison of Tables 2 and 4 show that the product compositions determined from simulation and experiment are in good agreement.

In addition to the final compositions presented in Table 4, we present the ratio of impurity in the intermediate vessels 2 and 3. The ratio of impurity is given by the concentration of lighter than key component ($x_l$) to the concentration of heavier than key component ($x_h$). The ratio of impurity gives an indication in which direction we have to change the temperature setpoint in the section adjacent to a vessel to achieve a certain product quality. We conjecture that the degree of separation is maximized when the ratio of the light and heavy impurities, $\frac{x_l}{x_h}$, in the intermediate vessels is close to 1.

The number of stages in the experimental system, depending on the reboiler duty, is determined from pseudo-binary vapor-liquid equilibrium data to be $N_{T,1} = 8 \pm 1$ in column section 1, $N_{T,2} = 11 \pm 1$ and $N_{T,3} = 7 \pm 1$ in section 2 and 3 respectively. A rather low number of stages is recorded for experiments 2 to 4, as indicated by the rather low product purities of the intermediate vessels, this is caused by operation close to flooding. Further, the different number of stages in the sections is caused by the slightly different volumetric vapor and liquid load in the sections (Note: we manipulate the volumetric flow rates in the experiments whereas molar flow rates are used in the simulation. However the temperature setpoints $T_{s,i}$ are identical). The experimental results in Table 4 show good agreement with the simulations presented in Table 2. An important reason for the observed deviations is that we used an integer number of stages in each section in the simulation study whereas packed column sections are used in the experiment.

Table 4: Summary of initial feed composition and main component in final products; temperature setpoints in all cases are $T_{s,i} = [71.5^o, 87.75^o, 107.2^o]$

| Exp. | reboiler duty $[\frac{J}{s}]$ | date | feed composition $z_F$ | product composition $x_1(M_1)$ | $x_2(M_2)$ | $x_3(M_3)$ | $x_4(M_4)$ | impurity ratio $\frac{x_1}{x_3}(M_2)$ | $\frac{x_2}{x_4}(M_3)$ |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 350 | 27.nov.'95 | [.24, .22, .21, .33] | 0.982 | 0.960 | 0.924 | 0.947 | 0.57 | 1.28 |
| 2 | 450 | 06.des.'95 | [.26, .18, .16, .40] | 0.940 | 0.886 | 0.884 | 0.934 | 4.39 | 11.79 |
| 3 | 380 | 23.mar.'96 | [.27, .19, .20, .34] | 0.928 | 0.912 | 0.909 | 0.985 | 2.50 | 5.91 |
| 4 | 390 | 03.apr.'96 | [.20, .15, .21, .44] | 0.936 | 0.919 | 0.907 | 0.993 | 8.83 | 9.83 |
| 5 | 375 | 24.sept.'96 | [.18, .13, .10, .59] | 0.978 | 0.915 | 0.962 | 0.925 | 7.50 | 0.52 |
| 6 | 385 | 01.oct.'96 | [.12, .13, .14, .61] | 0.969 | 0.937 | 0.950 | 0.959 | 3.50 | 0.67 |
| 7 | 370 | 04.oct.'96 | [.40, .04, .07, .49] | 0.971 | 0.922 | 0.945 | 0.961 | 5.00 | 0.35 |
| 8 | 380 | 17.oct.'96 | [.17, .16, .16, .51] | 0.960 | 0.929 | 0.941 | 0.961 | 4.92 | 0.67 |
| 9 | 375 | 18.oct.'96 | [.20, .15, .15, .50] | 0.963 | 0.923 | 0.941 | 0.966 | 3.76 | 0.74 |
| 10* | 350 | 19.oct.'96 | [.18, .15, .14, .53] | 0.969 | 0.914 | 0.933 | 0.962 | 5.11 | 0.69 |
| 11* | 355 | 20.oct.'96 | [.18, .15, .14, .53] | 0.970 | 0.931 | 0.939 | 0.958 | 3.60 | 0.59 |
| 12 | 360 | 07.nov.'96 | [.26, .12, .18, .44] | 0.971 | 0.931 | 0.945 | 0.949 | 5.18 | 0.81 |
| 13 | 370 | 18.nov.'96 | [.18, .16, .16, .52] | 0.963 | 0.924 | 0.937 | 0.957 | 3.97 | 1.27 |
| 14* | 360 | 19.nov.'96 | [.18, .16, .14, .52] | 0.972 | 0.928 | 0.933 | 0.967 | 3.55 | 0.73 |

*Note: The liquid was initially charged to the reboiler vessel, except for the experiments marked with $*$ where the feed was initially distrubuted to all four vessels.*

In Figure 4 we present the controlled temperature profiles (top) in the vessels 1 to 4 and temperature transients in the column sections (bottom). Figure 5 shows the transient of the reboiler heat input (top) and the reflux flows (bottom) to the column sections. To enable the comparison between simulation and experiment, it is necessary to consider the time to heat up the column holdup. This is done by changing the time axis such that $t = 0$ is defined where $L_i = 0$ initially.

The start-up and operation of the column is explained by referring to Figures 4 and 5 and is as follows: The feed charge is filled to the reboiler and heated to its boiling point by an electrical heater. The boiling point of the feed mixture is reached at ($t = -0.3\ h$) and vapor rises through the column sections (see Figure 4 bottom, at $t \geq -0.1h$). The vapor is condensed and collected in the accumulator. When liquid starts collecting in the uppermost vessel ($M_1$), the reflux flow controllers $TC_i$ are activated at $t \geq 0h$ and reflux $L_i$ is recycled to the column sections. Simultaneously, the setpoint to the reboiler heater is reduced to a prespecified value (*e.g.:* $\Delta T = 120^oC$), the temperature difference between reboiler holdup and heating mantle is kept constant to control the reboiler duty. All reflux flow controllers are activated simultaneously to establish liquid flow downwards the column, thus emptying the reboiler and accumulation of light component in the lower vessels is avoided. Additionally, an override on the reflux controller $TC_2$ and $TC_3$ is implemented, to assure that the volumetric flows of $L_2 = L_3 \geq 1.1\ L_1$ during start-up. This is necessary, since the relation between composition and temperature for a multicomponent mixture is non-unique during start-up of the column. As time increases, a pseudo-binary separation is performed in all column sections and a unique solution of the composition-temperature relation will be possible.

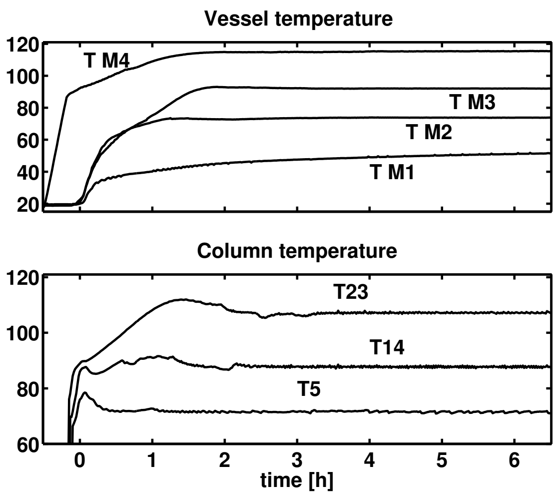


Figure 4: Temperature responses, experiment 10

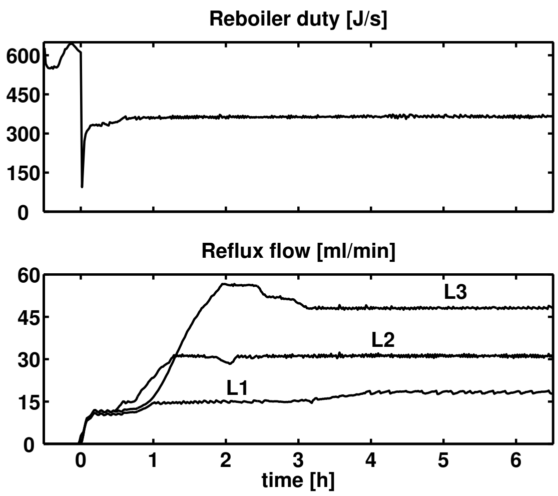


Figure 5: Manipulated variables, experiment 10

For $t \geq 1h$, the reflux flow controllers manipulate the flows such that setpoints $T_{S,5}$, $T_{S,14}$ and $T_{S,23}$ are approached. The control action of the reflux flow controls indirectly the level in vessels $M_1$ to $M_3$. Distillation is continued for a pre-specified time after all setpoints are approached to achieve some desired product compositions in the vessels.

In Figures 6 and 7 the time responses of vessel temperature, column temperature, reboiler effect and reflux flows recorded from experiment 7 are presented. From Table 4 we see that the amount of intermediate boiling components in experiment 7 is much less than in experiment 10, such that a rather low holdup in the intermediate vessels is expected. Due to the large amount of light component present in the column, temperature $T_5$, in the uppermost column section (see Figure 6, bottom) was initially so low that no reflux was recycled to the column. To avoid draining of the reboiler, reflux was therefore set manually in the time interval $0 \leq t \leq 1\ h$. Later, after accumulating a considerable amount of light component in vessel 1, temperature $T_5$ increased and the control system performed satisfactory and the setpoints in the column were approached.

The compositions of the main component in the vessels and the most important impurities from experiment 2 are shown in Figure 8. Comparing the trajectories of the main components in the vessels (Figure 8, top) with the simulation (Figure 2a), we see that the trajectories are similar in shape for the purification of the lightest component (1) in vessel $M_1$ and the heaviest component (4) in the reboiler $M_4$. The intermediate components (2 and 3) accumulated in vessel 2 and 3 are purified at a slightly slower rate, due to a lower number of stages and a significantly lower boilup ratio of the experiment compared to the simulation (compare Table 1 and 3).

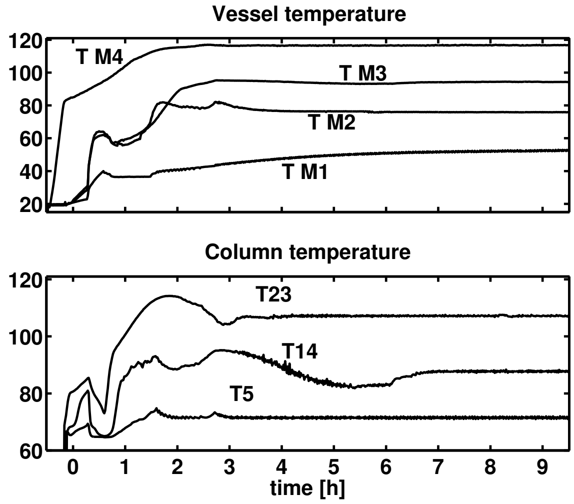


Figure 6: Temperature responses, experiment 7

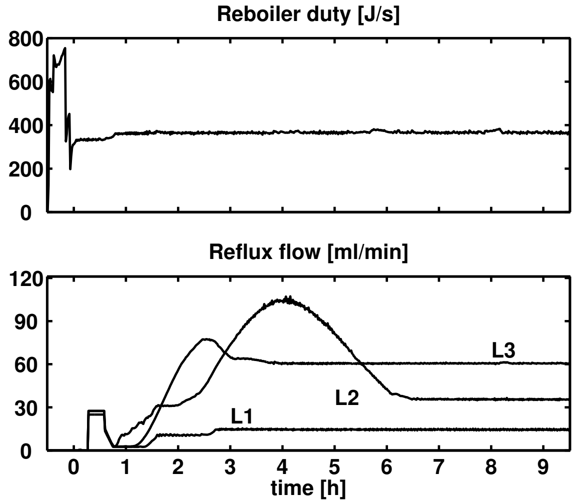


Figure 7: Manipulated variables, experiment 7

In Figure 9 we present the steady-state column temperatures from simulation (—) superimposed by the measured temperature (*) from experiment 10 given in Figure 4, we find good agreement between experiment and simulaton. Further, we see that a temperature profile similar to a distillation column separating a binary mixture, is developed in each of the three column sections.

# DISCUSSION

One justification for using multivessel distillation instead of conventional batch distillation is to save energy, or equivalently, for a given heat input the batch time may be significantly shorter. Another advantage is the simple operation of the multivessel column under total reflux. A third advantage is that the column operates close to an optimum separation because of the total reflux operation mode. In conventional batch distillation the optimal operation may depend on the reflux policy and quite strongly on the use of off-cuts to achieve the desired product composition, whereas in the multivessel batch column there are fewer degrees of freedom and this simplifies the operation. The reflux flow is adjusted with simple temperature controllers such that the desired products are accumulated in the vessels.

A disadvantage with the multivessel column compared to 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 multivessel columns is therefore more closely linked to a specific feed mixture, in particular the relative volatility and the product specifications. Thus, the design process of a multivessel column is similar to the design of a sequence of continuous distillation columns.

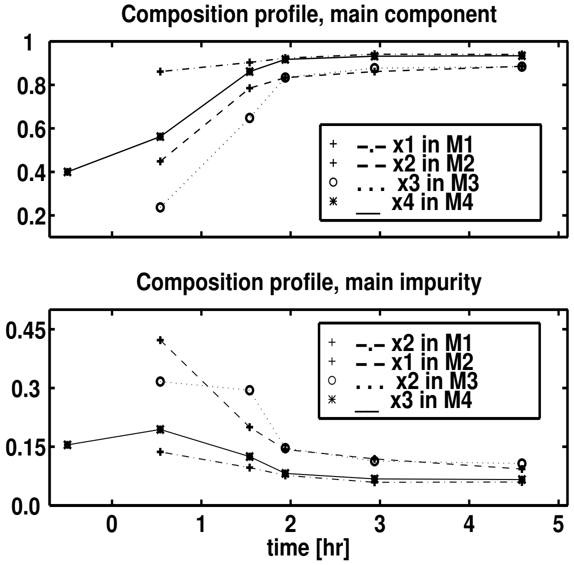


Figure 8: Compositions of main components (top) and the largest impurity (bottom) determined from experiment 2

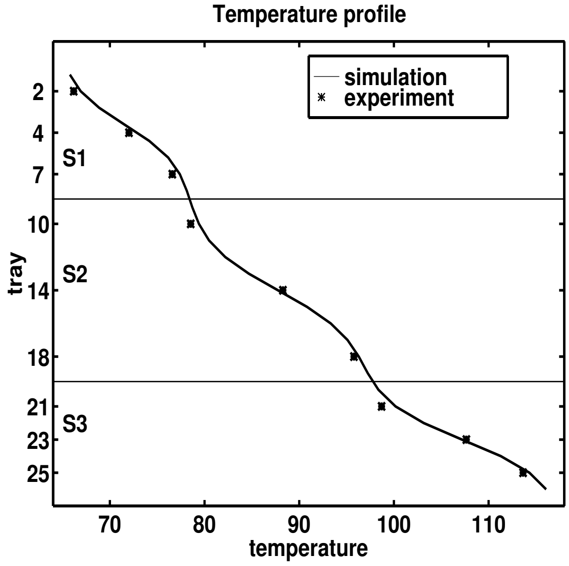


Figure 9: Temperatures in the column sections, determined from experiment 10 (*) and simulation (__)

A simple practical implementation, which is used in our laboratory scale column, is to place the sections and stages on top of each other as indicated in Figure 3. The liquid then flows by the influence of gravity and there is no need for pumps. However, this design is rather inflexible, and it cannot be used if a large number of stages is required. For an industrial multi-purpose separation facility, it is probably better to place the column sections in series with the vessels at ground level as indicated by Hasebe *et al.* (1995). Reflux pumps are then needed to bring the liquid from the vessels to the column sections. In this case, one can quite easily put several column sections in series to meet the separation requirements for a given feed mixture. This arrangement increases the flexibility of the process with respect to the number of components to be separated, such that the flexibility approaches that of is a conventional batch distillation column.

Although the results presented in this paper on the temperature controlled multivessel column are most encouraging, a number of questions are open for further research.

1. In this study the setpoints for the temperature controllers were set such that the temperature in the middle of the section should equal the average of the boiling points of the components separated in that section. In general, this is not optimal, especially if the requirements for product purities are very different.

2. Reasonable criterions for aborting the total reflux operation should be established, that is, when is the improvement in product purity too slow to justify further operation, and how should this be detected.

3. Finally, the total reflux operation may be generalized by also allowing withdrawal of products (continuous or discontinuous) from the vessels. In this way the multivessel column forms a "super structure" which has as special cases all the previously proposed batch schemes mentioned in the introduction.

## CONCLUSIONS

The experiments show very good agreement with the simulations, and confirm that the multivessel column can be easily operated with simple temperature controllers, where the holdups are only controlled indirectly. For a given set of temperature setpoints, we confirm that the final product compositions are independent of the initial feed composition.


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## NOTATION

| | | | | | |
|---|---|---|---|---|---|
| $K_c$ | Controller gain $\frac{kmol}{h^oC}$; $\frac{ml}{min^oC}$ | $L$ | Reflux flow $\frac{kmol}{h}$; $\frac{ml}{min}$ | $M$ | Holdup $kmol$, $l$ |
| $N_c$ | Number of components | $N_i$ | Number of stages in section $i$ | $t$ | time $h$ |
| $T$ | Temperature $^oC$ | $T_b$ | Boiling temperature $^oC$ | $TC_i$ | Temperature controller |
| $V$ | Vapor flow $\frac{kmol}{h}$ | $x$ | Liquid composition | $y$ | Vapor composition |
| $z_F$ | Feed composition | $\alpha$ | Relative volatility | | |
| $i$ | section identifier | $j$ | component identifier | | |