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Experimental and Theoretical Studies on the Start-Up Operation of a Multivessel Batch Distillation Column

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Multivessel batch distillation is a promising alternative to conventional batch distillation. Earlier studies proved the feasibility of temperature control in a closed operation mode, that is, when no product is removed until the end of the process. In this article, the influence of the reflux strategy during the initial start-up period is investigated in detail. This is a very important task in terms of the development of automation procedures in an industrial realization. The work is divided into three main parts. First, a rigorous dynamic model is introduced to describe the behavior of a multivessel batch distillation during start-up. Then, two experiments carried out on a laboratory scale column were chosen to validate the model. Finally, a sensitivity study is presented observing composition response while changing the reflux strategy. The results show that the process can be improved by applying a high reflux ratio and establishing the hydraulics as soon as possible.

Introduction

Multivessel batch distillation is a superstructure of all batch distillation configurations. Mostly, the term is used for processes with at least four product vessels including the reboiler and the distillate receiver. In closed operation mode, no product is removed; that is, if no intermediate heating is applied and at fixed pressure, the process offers four degrees of freedom: reboiler duty and liquid reflux streams from the product vessels (Figure 1).

Multivessel batch distillation was the subject of several investigations in the past two decades.[1−4] The main objective was to prove the feasibility of the process and to investigate different process control strategies. Temperature control as proposed by Wittgens et al.[2] is especially favorable since the final product composition is independent of the feed composition. The temperature of the section can be easily used as a controlled variable. The set points are set to the arithmetic mean value of the boiling temperatures of the two key components to be separated in a column section. Besides remarks on alternative process control strategies, Wittgens and Skogestad[4] also commented on the start-up period based on experimental experiences. The authors recommend using an “override” to guarantee reflux during start-up in terms of feasibility; i.e., one should avoid emptying the reboiler by setting $L_3 \geq L_2 \geq L_1$ on a volumetric basis in this specific case. Furlonge et al.[6] performed dynamic optimization studies of a multivessel batch distillation column but concluded that no general guidelines on how the column should be run optimally can be given. The authors suggest carrying out optimization studies for each new case, which is, obviously, very time-consuming. Gruetzmann and Fieg[7] analyzed optimization potentials in a middle vessel batch distillation by varying the reflux strategy before temperature control is activated. The authors stated that early manipulations can decrease the process duration. Therefore, it is logical to have a closer look at the start-up period of a multivessel batch distillation column and conclude more general remarks. Thus, this article presents a reasonable extension of the previous work.

The article is divided into sections as follows. First, a definition of the term “start-up” is given and appropriate ways to automate this period are discussed. A mathematical model is presented that is capable of dealing with the start-up process. Then, the model is validated by comparison with two experiments chosen from a series of experimental studies. Finally,

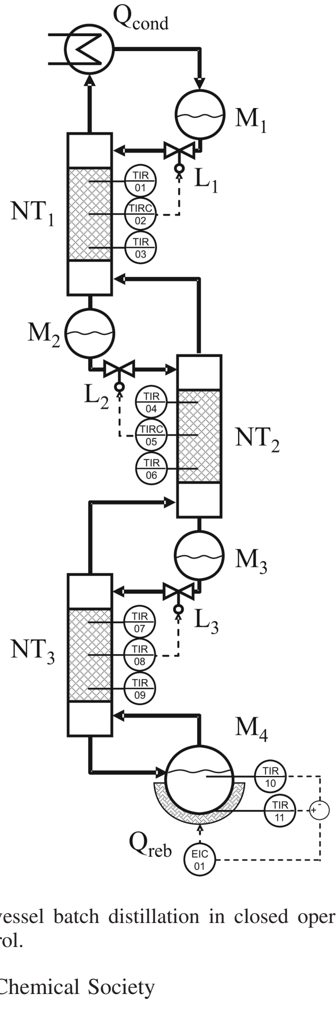

Figure 1. Multivessel batch distillation in closed operation mode using temperature control.

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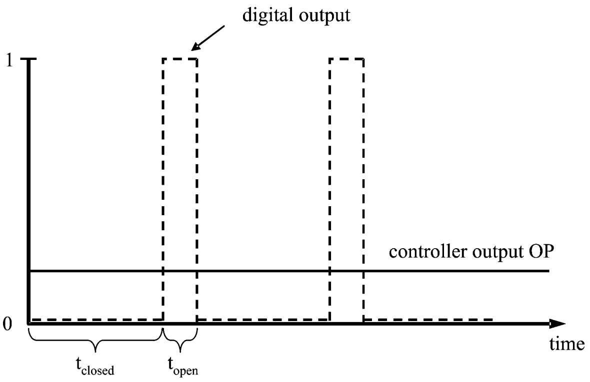

Figure 2. Modulation of the controller output.

the model is used to investigate the sensitivity of relevant parameters on the process duration.

Start-Up Operation

Start-up of continuous distillation usually refers to the period in which the column is transferred from an initial state at ambient conditions to a steady state operation mode.[8] During start-up of conventional batch distillation the column is heated and stable hydraulics are established. Afterward, the predefined operation mode (reflux strategy) is applied. Thus, the start-up period is defined as the period before any products are withdrawn. Considering a multivessel batch distillation in closed operation mode, that is, filling of the product vessels is inherent to the system, start-up is defined as the period in which the temperature controllers are inactive. This period includes (i) heating and vaporizing of the feed mixture, (ii) condensing of the vapor, and (iii) filling of the product vessels with an (optimized) reflux strategy. After start-up, temperature controllers lead the process to steady state. The main question then is how to choose the reflux ratio to fill the vessels optimally in terms of process duration without performing time-consuming optimization studies in advance. Two general ideas can be identified. One is to purify the column sump as fast as possible, while the other strategy is to establish reflux as soon as possible to guarantee separation in each column section. Due to the strong interconnections between the vessels, in particular vessels 2 and 3, finding the optimum strategy is not trivial, as confirmed by Furlonge et al.[6] Following the approach used by Wittgens and Skogestad,[5] we introduce the so-called "override" parameter denoted by a_k to investigate the column behavior during start-up. The parameter connects the volumetric liquid reflux streams from two adjacent vessels. It is defined by

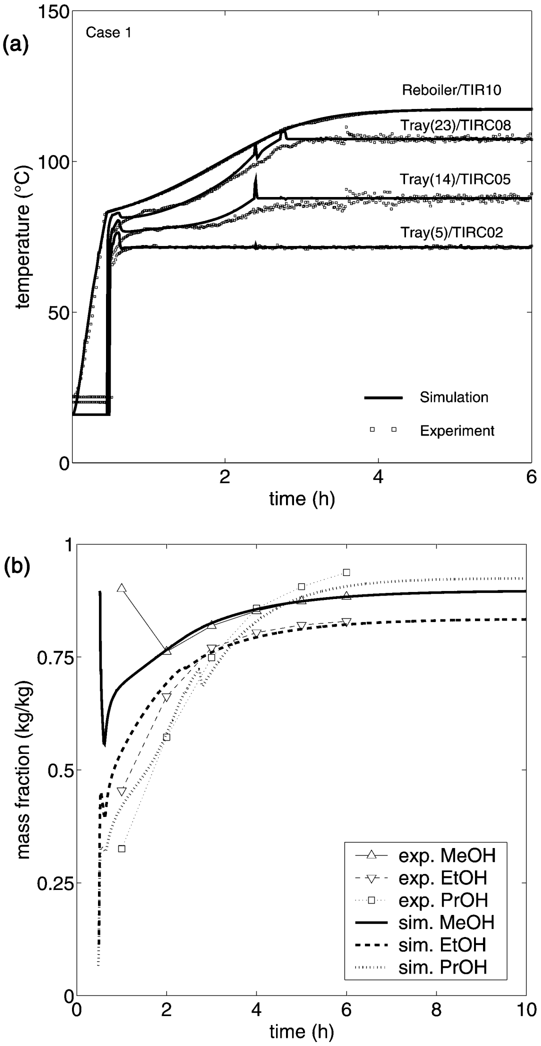

Figure 3. Comparison of experiment and simulation (case 1).

$$a_k \equiv \frac{L_k}{L_{k-1}}; \qquad k \in [2, 3] \tag{1}$$

where L_{k-1} denotes the reflux stream from vessel $k - 1$ that is located above vessel k (Figure 1). Then, the start-up proceeds

Table 1. Experimental and Simulation Setup

parameter	simulation	experiment
column diameter	30 mm	30 mm
type of packings	−	3 × 420 mm Raschig rings
number of theoretical stages	27	−
location used for temperature control	5/7/6	middle of each section
temperature set points	71.5/87.75/107.45 °C	71.5/87.75/107.45 °C
heat losses	approximately 4 W/stage (calcd)	−
feed mixture	MeOH, EtOH, *n*-PrOH, *n*-BuOH	MeOH, EtOH, *n*-PrOH, *n*-BuOH
feed charge	2.4 kg	2.4 kg
feed composition case 1	15/22/28/35 wt %	15/22/28/35 wt %
feed composition case 2	8.5/22/30.5/39 wt %	8.5/22/30.5/39 wt %
reboiler duty	approximately 300 W	approximately 300 W
top pressure	101.3 kPa	101.3 kPa
initial temperature	16 °C	16 °C
liquid reflux L_1 (during start-up)	100 mL/min (nominal)	100 mL/min (nominal)
controller gain	−0.5 mL/min/K	−0.5 mL/min/K
integral time	2 min	2 min
override a_2; a_3 in case 1	1; 1	1; 1
override a_2; a_3 in case 2	0.8; 1	0.8; 1

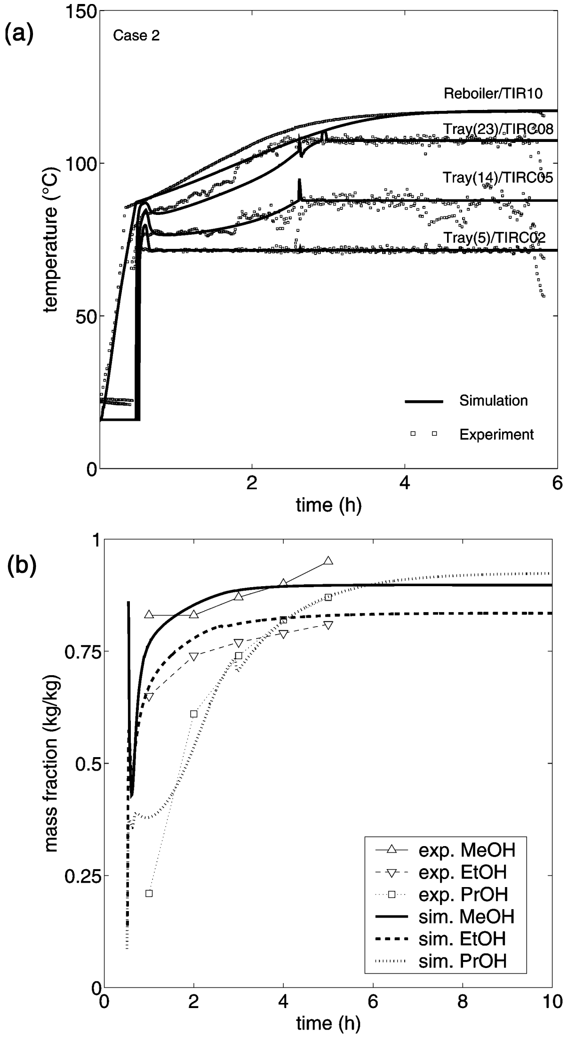

Figure 4. Comparison of experiment and simulation (case 2).

Table 2. Termination Criteria and Final Process Duration in Cases 1 and 2 (Simulation Data)

case	$t_{t,1}$	$t_{t,2}$	$t_{t,3}$	$t_{t,4}$	t_f
1	6.5	6.5	**6.7**	4.4	6.7
2	3.2	4.4	**7.2**	4.7	7.2

Table 3. Simulation Parameters

parameter	simulation
total number of theoretical stages	27
feed mixture	MeOH, EtOH, *n*-PrOH, *n*-BuOH
location used for temperature control	5/5/5
temperature set points	71.5/87.75/107.45 °C
feed charge	2.4 kg
composition	25 mol % (each)
reboiler duty	approximately 240 W
liquid reflux L_1 (during start-up)	0.24 kg/h
override a_2 and a_3	0/0.2/0.4/0.6/0.8/0.9/1/1.1

Table 4. Process Duration

a_2,a_3	$t_{t,1}$	$t_{t,2}$	$t_{t,3}$	$t_{t,4}$	t_f
1	3.24	3.16	**3.64**	3.09	3.64
0.9	3.28	**3.52**	3.51	2.65	3.52
0.8	3.29	**3.72**	3.51	2.46	3.72
0.6	3.31	**3.92**	3.65	2.30	3.92
0.4	3.33	**3.97**	3.75	2.22	3.97
0.2	3.34	**3.97**	3.85	2.16	3.97
0	3.35	**3.97**	3.92	2.13	3.97

as follows. First, the distillate reflux L_1 is defined, e.g., on the basis of the minimum liquid load to guarantee separation in the uppermost column section. Note that since we expect the purification of the products in vessels 2 and 3 to dominate the process duration, we did not use L_1 as a sensitivity parameter in the following sections. Second, liquid is accumulated in vessels 2 and 3 following eq 1 until the temperature control loops are activated. In the simulation, this is done when the temperature equals the set point for the first time. Stable hydraulic profiles develop afterward, and the process can be terminated in accordance with some predefined criteria. In this work, we use the approach to the steady state key composition in the product vessels to indicate the process duration.

$$t_{t,i} = t|_{x_{i,k} \geq 0.99 x_{i,k}^{\infty}} \tag{2}$$

$$t_f = \max(t_{t,i}) \tag{3}$$

where t_f denotes the final process duration, $t_{t,i}$ is the time to fulfill the termination criteria in vessel i, $x_{i,k}$ is the actual mass fraction of the key component i in vessel k, and $x_{i,k}^{\infty}$ is the steady state mass fraction determined by simulation.

Process Modeling

In this section the mathematical model used for sensitivity studies is presented. The model was originally used for a middle vessel batch distillation and is similar to the approach applied by Wang et al. for conventional batch distillation.[9,10] Two experiments chosen from a series of experimental studies at a multivessel batch distillation column were taken to prove the validity of the model in this case, in particular during start-up. The experiments differ in the feed composition and the start-up strategy.

The inherent change of state variables in batch distillation is typically highly nonlinear. The HETP value was used to set up a tray-to-tray equilibrium model in the modeling and simulation software Aspen Custom Modeler. In order to describe the start-up behavior accurately, the following features were included in the model:

- heating of the distillation plant
- heating of the feed
- filling of the theoretical trays
- variable relative volatilities
- ideal and nonideal vapor−liquid equilibrium
- variable molar vapor flow
- nonadiabatic operation

The vapor holdup on a tray has been neglected. The necessary physical and thermodynamic properties of the components as well as vapor−liquid equilibrium data are provided by internal procedures from Aspen Custom Modeler. Details can be found in Gruetzmann and Fieg.[11]

Material and Energy Balances.

$$\frac{d(M_j)}{dt} = L_{j-1} + V_{j+1} - L_j - V_j \tag{4}$$

$$\frac{\mathrm{d}(M_j x_{i,j})}{\mathrm{d}t} = L_{j-1}x_{i,j-1} + V_{j+1}y_{i,j+1} - L_j x_{i,j} - V_j y_{i,j} \tag{5}$$

$$\frac{\mathrm{d}(M_j h_{\mathrm{liq},j} + H_{j,\mathrm{internals}} + H_{j,\mathrm{shell}})}{\mathrm{d}t} = L_{j-1}h_{\mathrm{liq},j-1} + V_{j+1}h_{\mathrm{vap},j-1} - L_j h_{\mathrm{liq},j} - V_j h_{\mathrm{vap},j} - Q_{\mathrm{loss},j} \tag{6}$$

$$H_{j,\mathrm{internals}} + H_{j,\mathrm{shell}} = (mc_pT)_{j,\mathrm{internals}} + (mc_pT)_{j,\mathrm{shell}} \tag{7}$$

The heat capacities of the column internals and the shell are considered by mc_p with indices "internals" and "shell".

Summation Equations.

$$\sum_i x_{i,j} = 1 \tag{8}$$

$$\sum_i y_{i,j} = 1 \tag{9}$$

Vapor–Liquid Equilibrium.

$$y_{i,j} = K_i x_{i,j} \tag{10}$$

K denotes the equilibrium constant and is summoned from Aspen Properties depending on the chosen physical equilibrium method.

Tray Hydraulics. If the vapor pressure of the mixture $p_{\mathrm{VL},j}$ is larger than the pressure on the tray p_j, a vapor outlet stream V_j is computed.

$$V_j = \frac{p_{\mathrm{VL},j} - p_j}{C_1} \tag{11}$$

The constant C_1 was adjusted to reduce the difference between $p_{\mathrm{VL},j}$ and p_j without running into numerical problems. The liquid outlet stream is calculated in a similar way.

$$L_j = \frac{M_j - M_{\mathrm{max},j}}{C_2} \tag{12}$$

Here, $M_{\mathrm{max},j}$ denotes the maximum theoretical liquid holdup on a tray determined by empirical algebraic correlations. If the holdup on a tray exceeds $M_{\mathrm{max},j}$, an outlet flow is calculated. The parameter C_2 corresponds to the hydraulic time constant and has to be adjusted depending on the column that is investigated.

Reflux from Product Vessels. The overall process model consists of similar sets of equations to describe the reboiler, the condenser, and the product vessels. Parallel PI controller algorithms were used in both simulation and experiment to calculate the reflux from the product vessels. The controller output OP is calculated by eq 13. Here, SF denotes a scaling factor determined during the initial experiments. It equals the maximum flow through the valve. Since solenoid valves were used at the laboratory scale column, this parameter is necessary to provide a signal within the operating range of OP.

$$\mathrm{OP} = \frac{1}{\mathrm{SF}}\left(\mathrm{bias} + K_{\mathrm{p}}\varepsilon + \frac{1}{T_{\mathrm{i}}}\int \varepsilon\,\mathrm{d}t\right), \quad \mathrm{SF} = 6\ \mathrm{L/h}, \quad \mathrm{OP} = [0;1] \tag{13}$$

$$\varepsilon = \mathrm{SP} - \mathrm{PV} \tag{14}$$

Subsequently, a digital signal is generated by modulation as defined in eq 15 and illustrated in Figure 2.

$$t_{\mathrm{open}} = (\mathrm{OP})t_{\mathrm{cycle}} \tag{15}$$

The actual outlet flow in the experiment then depends on the opening time of the valve t_{open} and the liquid level in the product vessel. A cycle time t_{cycle} of 2 s was found out to work well.

In the simulation, the same continuous controller output OP was used to determine the actual liquid reflux as follows:

$$L_k = (\mathrm{OP})L_{\mathrm{max},k} \tag{16}$$

The dependency on the liquid level was considered by using Torricelli's formula (eq 17) to calculate the maximum outlet flow $L_{\mathrm{max},k}$; that is, the liquid flow that goes through the valve if it is always open.

$$L_{\mathrm{max},k} = A\sqrt{2gh_k} \tag{17}$$

Here, A denotes the cross-sectional area of the reflux pipe, g is the gravitational constant, and h_k is the liquid level. Referring to the experimental validation, the number of theoretical stages as well as the stages used for temperature control were adjusted in accordance with the steady state product qualities. They were numbered from the top to the bottom. The reboiler duty was determined by the time needed to heat the feed and the column during initial experiments. Heat losses were calculated by heat transfer correlations taken from the literature.[12] The model was initialized using an empty column at ambient conditions. The feed was supplied to the reboiler.

Experimental Setup. The laboratory scale column sketched in Figure 1 is made of glass and insulated with mineral wool to reduce heat losses. The reboiler offers a maximum volume of 4 L, while the other product vessels can contain 1 L. Each column section has a diameter of 30 mm and a height of 420 mm filled with stainless steel Raschig rings (3 × 3 mm). Heating was supplied by a heating mantle with a maximum heat duty of 687.5 W. While the temperature in the reboiler increases during the process, a (constant) temperature difference between the wall and the liquid was used as a controlled variable to maintain the reboiler duty. A temperature profile over the entire column height was recorded using three thermocouples placed in each section as sketched in Figure 1. Solenoid valves operated by solid state relays were used to control the reflux from the product vessels. Temperature measurements, solenoid valves, and heating tapes are connected to a FieldPoint control system by National Instruments. LabVIEW was used for data acquisition and process control with a sampling frequency of 0.5 Hz. A fairly ideal zeotropic mixture of methanol, ethanol, *n*-propanol, and *n*-butanol was separated into four fractions. The column was operated at atmospheric pressure.

The temperature in the middle of each section was used to control the reflux from the product vessels. In accordance with Wittgens et al.[2] the set point T_{SP} is then calculated following

$$T_{\mathrm{SP},k} = \frac{1}{2}(T_{\mathrm{b},k} - T_{\mathrm{b},k+1}) \tag{18}$$

with $T_{\mathrm{b},k}$ and $T_{\mathrm{b},k+1}$ as the boiling temperature of the light and heavy key components to be separated in column section k. Standard PI controllers were adjusted to avoid extensive control actions during start-up. The experimental setup is summarized in Table 1.

Results. The results are displayed in Figures 3 and 4 for cases 1 and 2, respectively. Figures 3a and 4a show the temperature responses of chosen locations, while the mass fractions of the key components in vessels 1–3 are displayed in Figures 3b and

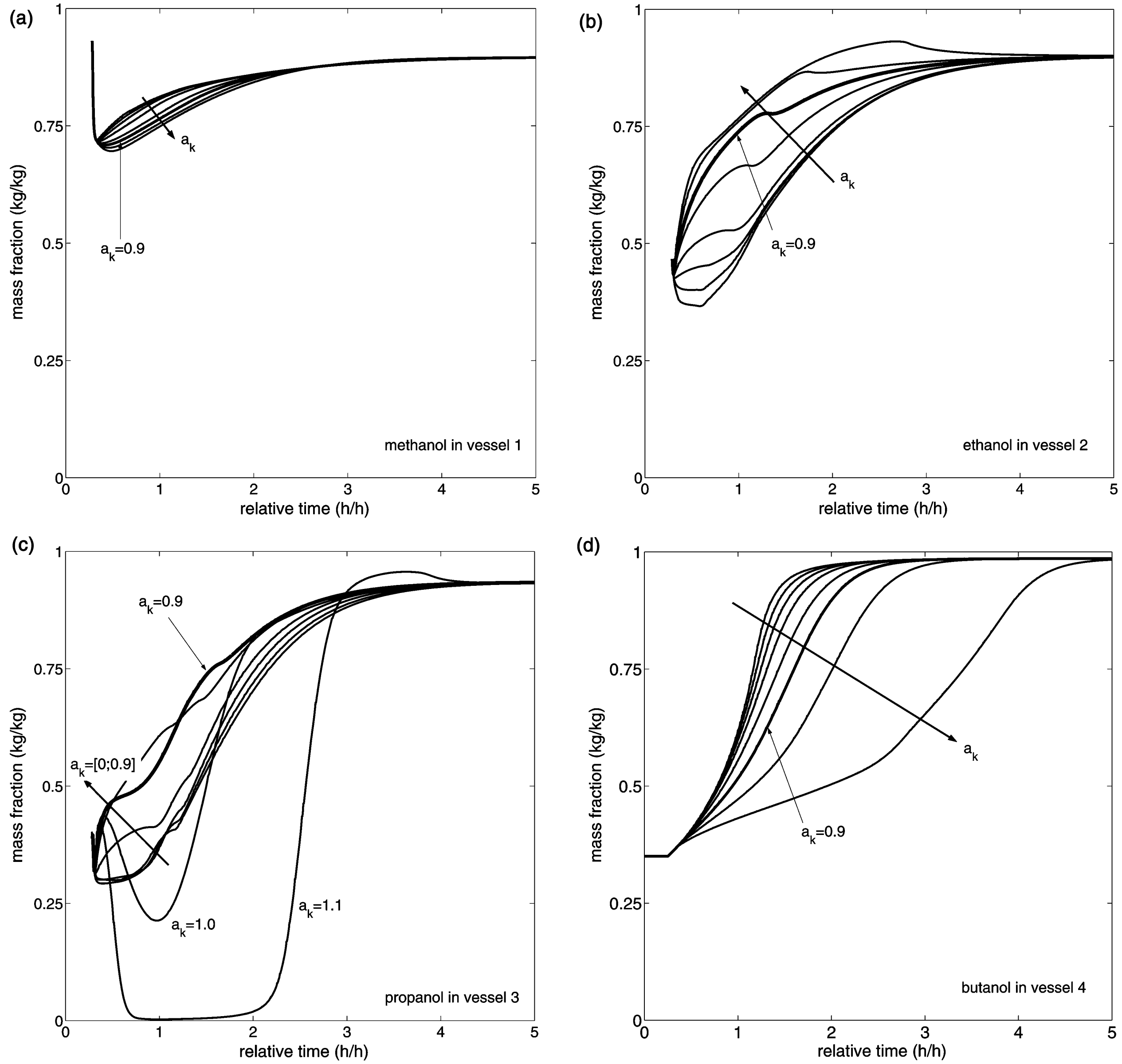

Figure 5. Response of the key components as a function of parameters a_k.

4b. The results clearly prove the validity of the model. Simulation and experiment agree quite well. Slight deviations can be explained by the assumptions named above and the discretization of the column sections.

A comparison of both experiments in terms of start-up behavior and process duration is difficult because of the different feed compositions. However, some interesting facts can be summarized. First, the steady state composition is independent of the feed composition using temperature control; that is, the composition in the product vessels tends to be the same in both experiments. This confirms the results of Wittgens et al.[2] Second, a small decrease of a_2 from 1 to 0.8 leads to clear changes in the process behavior. The process duration of the first case indicated by an achievement of 99% of the steady state key mass fraction is approximately 6.7 h, while in the second case it is approximately 7.2 h. In both cases, *n*-propanol is the limiting key component. However, the time to meet the termination criteria of the other key components clearly differs between the two cases, as summarized in Table 2. A detailed study of the influence of the override on the process is the objective of the next section.

Simulation Studies

Based on the presented results and information obtained from further experimental studies, a sensitivity study was carried out to investigate the influence of the override parameter a_k on the process duration. The simulation setup is summarized in Table 3. The reflux is independent of the liquid level. This is in accordance with the industrial practice where reflux pumps are used. The override is connected to the volumetric flows and is varied between 0 and 1.1 until temperature control is activated. The lower and upper bounds were chosen to cover the entire range of feasibility. If $a_k = 0$, no reflux is sent back to the column from vessels 2 and 3. Then, separation is only efficient in the lower parts (i) if the internal liquid flow caused by condensation is high enough or (ii) after reflux is established by temperature control. Since the temperature in the column increases with the amount of feed evaporated, this strategy basically works. However, in some scenarios problems may arise

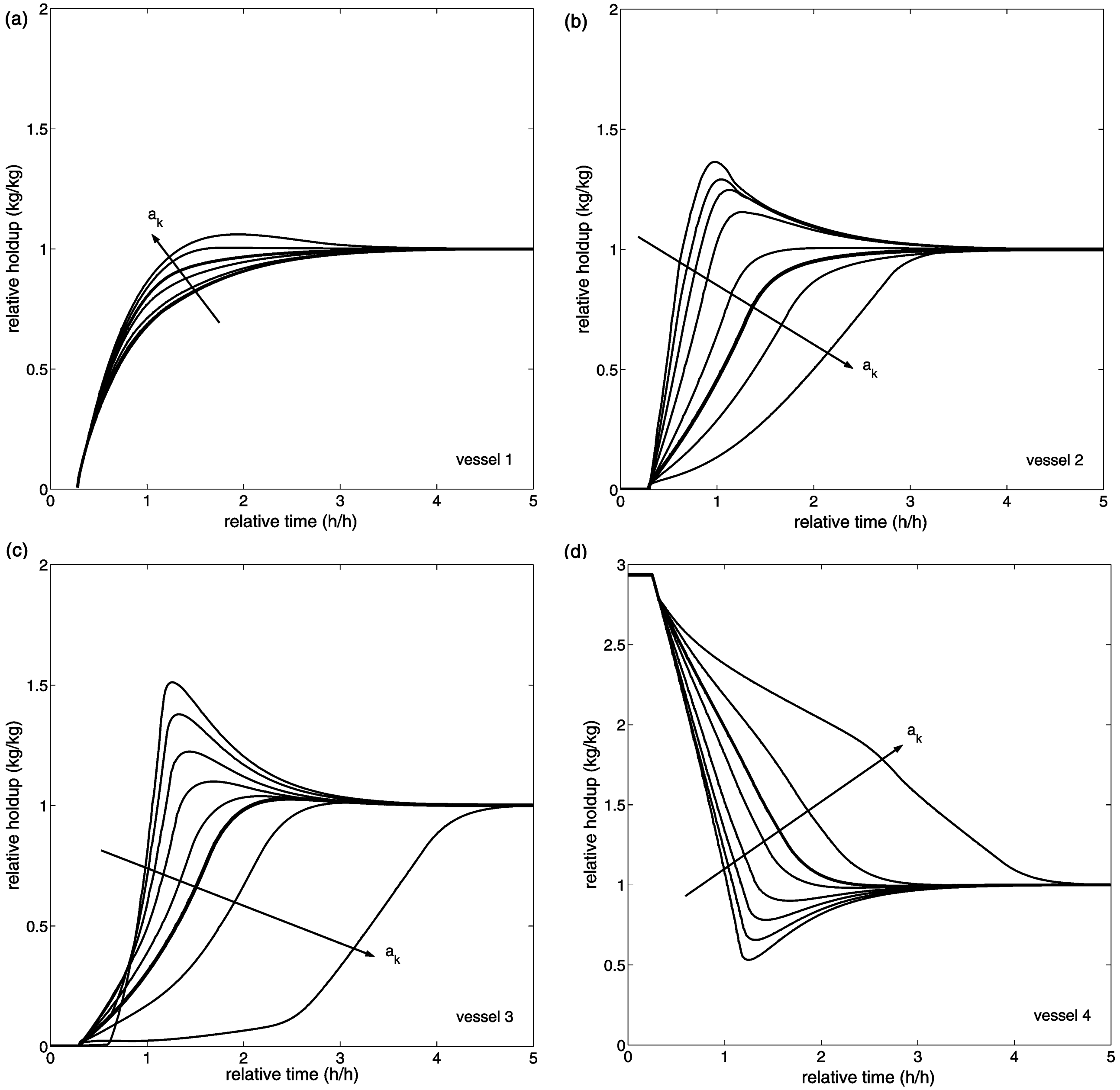

Figure 6. Response of the holdups as function of parameters a_k.

due to the fact that there is no unique relationship between temperature and composition during start-up. Note that in the other scenario that $a_k = 1.1$, and it is still possible to accumulate liquid because the volumetric flow increases from the top to the bottom. This is due to an increase in the molecular weight from methanol to *n*-butanol. In other cases it may be necessary to redefine the upper bound of a_k.

Results and Discussion

Table 4 summarizes the time to meet the termination criteria for each key component $t_{t,i}$ and the final process duration t_f. Figure 5 illustrates the mass fractions of the key components as a function of the relative process time, which is defined as the ratio between the actual process time and the time to reboil the initial feed (eq 19).

$$\tau = \frac{t}{M_{4,0}/V} \tag{19}$$

Figure 6 shows the development of the relative holdup in all vessels. The relative holdup is defined as the actual holdup divided by the final holdup (eq 20).

$$\mu_k = \frac{M_k}{M_{k,f}} \tag{20}$$

It can be clearly seen from Table 4 that an increase in a_k from 0 to 0.9 leads to a decrease in process time. The purification of ethanol in vessel 2 is limiting. The reason is that "impurities", indicated by the undershooting of the composition response in Figure 5b, accumulate in vessel 2 temporarily. Interestingly, the amount of both main impurities, methanol and *n*-propanol, is responsible for the delay. If a_k is further increased above 0.9, the process is slowed down. Since the sump is depleted very slowly, if we approach the upper bound of a_k, this behavior is expected. It can be further observed that the limiting key component changes with an increase in the override. First, the limiting key component is ethanol, while it is

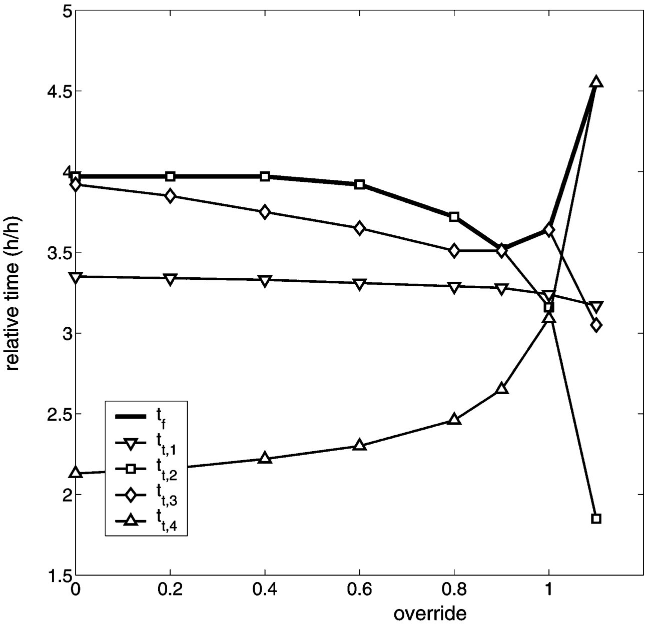

Figure 7. Limiting key component and process duration.

n-propanol if $a_k = 1$. The minimum process time can be observed when the purification in both vessels 2 and 3 needs approximately the same time to meet the termination criteria, as illustrated in Figure 7. The thin lines indicate when the products reach the termination criteria as a function of the override. The process duration, always given by the slowest "purification process", is illustrated by the thick line. The minimum duration for $a_k = 0.9$ can be clearly seen. The a priori prediction of this value is subject to future work. However, establishing stable hydraulics as soon as possible improves the process significantly. Furthermore, one should avoid using low values of a_k. As Figure 6a,b indicates, the steady state holdup is exceeded if a_k is chosen to approach 0. This can lead to operational problems if the intermediate vessels are poorly designed.

On the basis of the presented results, we recommend operating the multivessel column with a high reflux ratio. However, it is important to notice that the use of a_k depends on the unit of measurement. In practice, valves and pumps are installed at industrial columns and using molar units is only applicable in simulation studies. Therefore valid values of a_k must be reevaluated for each individual case. For any separation task in which the mass and volumetric flows increase from the top to the bottom, e.g., for homologous series of alcohols as investigated in this article, one can set $a_{k,\max} = 1$ without running into operational problems. In other cases, for example, if the molecular weight decreases from the light to the heavy boiler, this may give inappropriate values of the override, causing the vessels to never accumulate liquid.

Summary and Conclusion

This article deals with the start-up of multivessel batch distillation. The task is of high complexity since the process is highly nonlinear and the vessels are strongly interconnected. The objective was to perform sensitivity studies to better understand the process and gather experiences that support the development of automated start-up procedures. Therefore, a mathematical model, originally set up to describe a middle vessel batch distillation, was validated by comparison with experiments. The experimental studies were carried out at a laboratory scale column. The results showed that simulation and experiment agree very well. The idea of the sensitivity study was to couple the refluxes from the product vessels by using an override parameter. Indeed, the results showed an effect of the reflux ratio on the process duration. Interesting results concerning the feasibility and performance of the strategies were obtained. These will serve as basis for further studies to determine the optimal reflux and draw more general conclusions. However, it seems clear that the process is improved by applying a high reflux ratio and establishing the hydraulics as soon as possible. We can conclude that the override provides a useful support to automate the start-up procedure.

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Symbols

a = override parameter
A = cross-sectional area (m^2)
c_p = specific heat capacity (J kg^{-1} K^{-1})
C = constant
g = gravitational constant (m s^{-2})
h_k = liquid level (m)
h = specific enthalpy (J mol^{-1})
H = enthalpy (J)
K = equilibrium constant
K_p = proportional gain (mL min^{-1} K^{-1})
L = liquid flow (mol s^{-1}; m^3 s^{-1}; kg s^{-1})
M = holdup (mol)
NT = number of theoretical stages
p = pressure (Pa)
Q = heat duty (W)
t = time (s)
T = temperature (K)
T_i = integral time (min)
V = vapor flow (mol s^{-1})
x = liquid fraction (mol/mol)
y = vapor fraction (mol/mol)

Subscripts

b = refers to boiling temperature at given pressure
closed = closing time of the reflux valve
cond = condenser
cycle = cycle time
equipment = column internals and column shell
f = final
i = component index
internals = column internals, i.e., packings, accumulator, distributor
j = tray index
k = vessel index, column section index
liq = liquid
loss = refers to heat transfer to the surroundings
max = maximum
open = opening time of the reflux valve
reb = reboiler
shell = column shell
vap = vapor
VL = vapor−liquid, refers to vapor pressure

Superscript

∞ = steady state

Abbreviations

HETP = height equivalent to a theoretical plate (m)
MeOH = methanol
EtOH = ethanol
PrOH = *n*-propanol
BuOH = *n*-butanol
SF = scaling factor
SP = set point
OP = operating parameter, controller output
PV = process variable

Greek Symbols

ε = error
μ = relative holdup
τ = relative time

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