Start-up of Empty Cold Reactive Distillation Columns by means of Feedback Control Strategies: the Discontinuous Stage
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Start-up of Empty Cold Reactive Distillation Columns by means of Feedback Control Strategies: the Discontinuous Stage

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Abstract

A new approach for start-up of empty cold reactive distillation columns by means of feedback control strategies is presented. In this paper, the discontinuous stage of start-up is taken into account. This stage begins with the fill-up and heating process of the empty cold column and ends if vapour reaches the condenser. A corresponding model for the start-up process is developed. The control strategy is based on servo-control of the process variables temperature and liquid level of the reboiler with PI controllers and is combined with bottom product recycling. The proposed strategy is demonstrated and tested with the reactive distillation process of esterification of ethanol with acetic acid.

Keywords: reactive distillation, start-up, discontinuous stage, feedback control

1. Introduction

Reactive distillation (RD) processes integrate chemical reactions and distillation in the same process unit (Sundmacher and Kienle, 2003). This has several positive implications, for instance reduction of investment and operational costs and potential improvement of performance. However, the dynamic behavior of reactive distillation columns (RDC) is quite complex, especially during the start-up stage.

The start-up process of RDC's is of environmental and commercial importance, and represents an active field of research. For RD operation, start-up should be finished as fast as possible since all process variables are changing during this stage, and the product has to be disposed. For this purpose, suitable control strategies are needed. So far, there are only few publications addressing start-up of empty cold RDC, for instance Reepmeyer et al., 2003, 2004a, 2004b; Reepmeyer, 2004. In these papers, a start-up model is developed and open loop strategies for time optimal start-up are presented. In Reepmeyer, 2004, the open loop strategy is combined with mathematical optimization.

The motivation of this paper is to solve the problem of the complete start-up process of the cold empty column by means of feedback control strategies. Thus, an automatic start-up strategy is obtained. The feedback control method offers several important advantages when compared to the existing open loop strategies: for example the start-up process can be made insensitive against disturbances, operating safety can be improved, and a desired steady state can be reached exactly. However, a wide operating range has to be covered during start-up. Therefore, linearized models are inappropriate for the controller design.

The start-up process of an empty cold column is divided into four stages:

(I) fill-up and heating stage: feed tray and trays below are filled, reboiler is heated;
(II) after achieving boiling conditions the vapor rises through the trays and reaches the condenser;
(III) reflux of liquid into the column, trays above the feed are filled completely, end of this stage: in every tray all vapor and liquid flow rates exist, counter current liquid and vapor flows inside the whole column are reached;
(IV) dynamic process to reach the desired steady state.

The stages (I) and (II) can be merged to the discontinuous stage. In view of solving the control problem it is necessary to split the discontinuous stage into two stages. The third and fourth stage of start-up are called semi-continuous and continuous stage.

It is convenient to model different stages of the start-up procedure separately and to apply a different control strategy for each stage. Subject to the individual stages of start-up, four models and related control structures have to be developed. The outcome is a switching scheme consisting of several control policies based on different models.

In this contribution, we investigate the automatic start-up of the discontinuous stage, i.e. stages (I) and (II). In Sommer et al., 2006, the automatic start-up of stage (I) is presented separately. The discontinuous stage is relevant, because in most works on start-up of RDC's the trays are filled and warm.

A model is presented to describe the start-up process. A corresponding control strategy is presented based on servo-control of the process variables temperature and liquid level of the reboiler with PI controllers including anti-reset windup. The control strategy is combined with recycling partial bottom product back to the feed (bottom product recycling). The recycle can reduce the amount of waste during start-up.

Our start-up strategy has been tested for an example where ethyl acetate and water are produced from ethanol and acetic acid.

The paper is organized as follows. In the next section a model for the start-up process is presented. Accordingly, a control strategy is developed. The esterification plant is described in Section 4. The application and the test of our strategy are depicted in Section 5. A summary and an outlook are given in Section 6.

2. Modeling the Discontinuous Stage

In this section, we present a model to describe the dynamic behavior of the discontinuous stage of start-up of the RDC as shown in Fig. 1. The model is used to simulate as well as design the control loop. All symbols are applied in the following are listed and explained in Section 7. An equilibrium model and a kinetic description of the reaction have been chosen. The vapor hold-up is neglected. The model is built for homogeneous catalysis. The equations can be applied to the heterogeneous case with no effort. For each tray, one total mass balance, one energy balance and $nc-1$ component mass balances are formulated as follows.

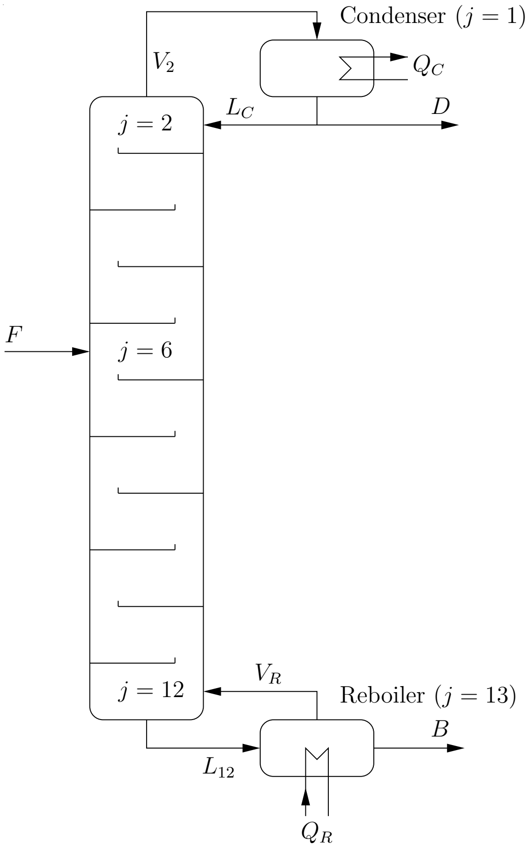

Figure 1: Ethyl acetate reactive distillation column.

Total Mass Balance:

$$\frac{dM_j(t)}{dt} = F_j(t)+L_{j-1}(t)-L_j(t)+V_{j+1}(t)-V_j(t)+\sum_{i=1}^{nc}\nu_i r_j(t)\varepsilon_j(t) \tag{1}$$

$$M_j(t) = \frac{A_j h_j(t)}{v_j^L(t)}, \quad v_j^L(t)=\sum_{i=1}^{nc} x_{i,j}(t) v_i^L \tag{2}$$

$$\varepsilon_j(t) = A_j h_j(t) \tag{3}$$

$$L_j(t) \;=\; \begin{cases} 0 & 0 \le h_j(t) \le H_{weir} \\ \dfrac{1.84 L_{weir}}{v_j^L(t)} \left(h_j(t) - H_{weir} \right)^{3/2} & h_j(t) > H_{weir} \end{cases} \tag{4}$$

Component Mass Balance:

$$\begin{aligned} \frac{d\left(M_j(t) x_{i,j}(t)\right)}{dt} &= F_j(t) x_{i,j}^F + L_{j-1}(t) x_{i,j-1}(t) - L_j(t) x_{i,j}(t) + \cdots \\ &\cdots + V_{j-1}(t) y_{i,j-1}(t) - V_j(t) y_{i,j}(t) + \nu_i r_j(t) \varepsilon_j(t) \end{aligned} \tag{5}$$

$$\sum_{i=1}^{nc} x_{i,j}(t) = \sum_{i=1}^{nc} y_{i,j}(t) = 1 \tag{6}$$

Energy Balance:

$$\begin{aligned} \frac{dE_j(t)}{dt} &= F_j(t) H_j^F - L_j(t) H_j^L(t) + L_{j-1}(t) H_{j-1}^L(t) + \cdots \\ &\cdots + V_{j-1}(t) H_{j-1}^V(t) - V_j(t) H_j^V(t) + Q_j(t) \end{aligned} \tag{7}$$

$$E_j(t) = M_j(t) H_j^L(t) + m_j c_{p,j} \left(T_j(t) - T_0 \right) \tag{8}$$

The balance equations have to be adapted for every special tray. The weir formula (4) was taken from Perry, Green, 1997 and the molar enthalpies of the liquid and the vapor phase are calculated by:

$$H_j^L(t) = \sum_{i=1}^{nc} x_{i,j}(t) H_{i,j}^L(t) = f(x,T)\,,\; H_j^V(t) = \sum_{i=1}^{nc} y_{i,j}(t) H_{i,j}^V(t) = f(y,T). \tag{9}$$

For trays inside the column, the tray geometry is illustrated in Fig. 2. The

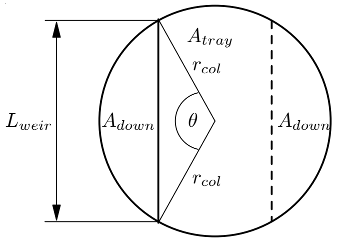

Figure 2: Tray geometry.

corresponding areas of each section are:

$$A_{col} \;=\; \frac{\pi}{4} d_{col}^2 = \pi r_{col}^2 \tag{10}$$

$$A_{down} \;=\; \frac{1}{2} r_{col}^2 (\theta - \sin\theta), \quad \theta = 2\arcsin\frac{L_{weir}}{2r_{col}} \tag{11}$$

$$A_{tray} \;=\; A_{col} - 2A_{down}. \tag{12}$$

The active area of the trays inside the column A_j is

$$A_j = A_{tray}, \tag{13}$$

and for the reboiler, we consider the same cross-section area as the column, i. e.

$$A_j = A_R = A_{col}. \tag{14}$$

The condenser is not considered, because the discontinuous stage is finished if vapor reaches the condenser.
During fill-up and heating no boiling conditions exist. For this reason, the vapor phase for modeling stage (I) of the start-up process does not occur ($V_j(t) = V_R(t) = 0$), and the pressure in the trays of the column are at initial pressure $p_j(t) = p_0$, for instance at ambient pressure. Stage (II) will start if boiling conditions are achieved in the reboiler and the model has to be switched. Vapor rises, and the trays above the reboiler are heated, such that the boiling conditions are reached in these trays step by step. Boiling conditions will be achieved in a tray (Reepmeyer et al., 2003) if the bubble pressure

$$p_{bub,j}(t) = \sum_{i=1}^{nc} x_{i,j}(t)\gamma_{i,j}(t) p_{i,j}^V(t) \tag{15}$$

is greater than the initial pressure:

$$p_{bub,j}(t) > p_0. \tag{16}$$

Then, the pressure in a tray $p_j(t)$ is set to the bubble pressure:

$$p_j(t) = \begin{cases} p_0 & p_{bub,j}(t) \le p_0. \\ p_{bub,j}(t) & p_{bub,j}(t) > p_0, \end{cases} \tag{17}$$

and the vapor and the liquid phase are in thermodynamic equilibrium:

$$y_{i,j}(t) p_j(t) = \gamma_{i,j}(t) p_{i,j}^V(t) x_{i,j}(t). \tag{18}$$

The vapor flow rate is calculated by:

$$V_j(t) = \begin{cases} 0 & p_j(t) \le p_{in,j}(t) \\ \dfrac{A_{holes}}{v_j^V(t)} \sqrt{\dfrac{2\left|p_j(t) - p_{in,j}(t)\right|}{\rho_j^V(t) c_w}} & p_j(t) > p_{in,j}(t), \end{cases} \tag{19}$$

with:

$$p_{in,j}(t) = p_{j-1}(t) + \rho_{j-1}^L(t) g h_{j-1}(t), \tag{20}$$

$$v_j^V(t) = \sum_{i=1}^{nc} y_{i,j}(t) v_{i,j}^V(t), \tag{21}$$

$$\rho_j^L(t) = \sum_{i=1}^{nc} x_{i,j}^{mass}(t) \rho_i^L, \quad x_i^{mass}(t) = \frac{x_i(t) MW_i}{\sum_{k=1}^{nc} x_k(t) MW_k}, \tag{22}$$

$$\rho_j^V(t) = \sum_{i=1}^{nc} y_{i,j}^{mass}(t) \rho_{i,j}^V(t), \quad y_i^{mass}(t) = \frac{y_i(t) MW_i}{\sum_{k=1}^{nc} y_k(t) MW_k}. \tag{23}$$

The molar volume v_i^L and the density ρ_i^L of the pure liquid components are regarded as constant. The molar volume $v_{i,j}^V(t)$ and the density $\rho_{i,j}^V(t)$ of the pure vapor components are functions of the temperature $T_j(t)$.

The generated model is a rather complex system consisting of implicit nonlinear differential equations. The implicit character of the model complicates the controller design. For the simulation of the implicit model, the commercial software package gPROMS (gPROMS Introductory User Guide, 2004) has been used.

3. Control Problem and Control Structure

First we focus on the control objective. During stage (I) the task is to fill-up the column and to heat the reboiler to achieve boiling conditions. In other words, the desired values of the liquid level h_R and temperature T_R in the reboiler should be controlled to reach the set points. Therefore, the controlled variables are the reboiler liquid level $h_R(t)$ and the reboiler temperature $T_R(t)$. During stage (II) the controlled variables should be kept constant. Possible manipulated variables are the feed flow rate $F(t)$, the bottom flow rate $B(t)$, and the reboiler heat duty $Q_R(t)$.

We propose to use $F(t)$ to control the reboiler liquid hold-up $h_R(t)$, and the reboiler heat duty $Q_R(t)$ to manipulate the reboiler temperature $T_R(t)$. The remaining input variable bottom flow rate is set to its steady state value: $B(t) = B_0$. With the manipulated variable flow rate $F(t)$, only this amount of feed is added to the column which is needed or which is necessary in every stage of start-up. During the steady state operation, the bottom flow rate $B(t)$ is a better choice to control the reboiler liquid level. Thus, a disturbance can be compensated faster. For the RDC we obtain a multi-input-multi-output (MIMO) system with two inputs and two outputs (Fig. 3). It is not reasonable, to control the mole fractions during the discontinuous stage, because no reflux and no distillate flow rate exist.

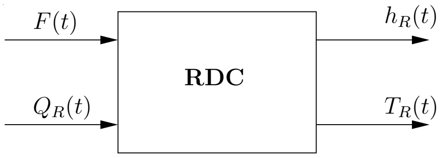

Figure3: Open loop structure of the RDC

We propose to use a PI structure to solve the control objective. For process stage under consideration $Q_R(t)$ influences $h_R(t)$ after vapor is rising. The feed flow rate $F(t)$ will have an impact on the reboiler temperature $T_R(t)$. However, two single PI control loops are attempted to apply. The control strategy is demonstrated in Fig. 4, and is described below. Two switching procedures are necessary during the automatic start-up of the discontinuous stage. Before the feed tray and the trays below, except the reboiler have been filled completely, the flow rate $F(t)$ is set to its maximum value F_{max} and the reboiler heat duty $Q_R(t)$ is set to zero. When the liquid arrives at the reboiler, the liquid level control and the temperature control are switched on:

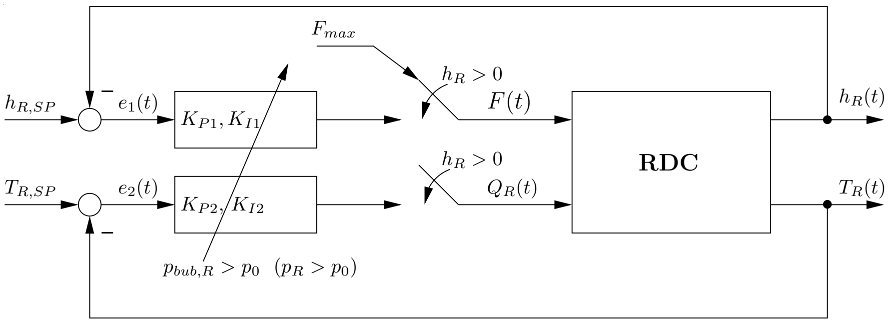

Figure 4: Control structure

$$u_1(t) = F(t) = \begin{cases} F_{max} & h_R(t) = 0 \\ K_{P1} e_1(t) + K_{I1} x_{I1}(t) & h_R(t) > 0, \end{cases}$$

$$u_2(t) = Q_R(t) = \begin{cases} 0 & h_R(t) = 0 \\ K_{P2} e_2(t) + K_{I2} x_{I2}(t) & h_R(t) > 0, \end{cases} \tag{24}$$

with the control errors:

$$e_1(t) = h_{R,SP} - h_R(t), \quad e_2(t) = T_{R,SP} - T_R(t), \tag{25}$$

and the state equations:

$$\dot{x}_{I1}(t) = \begin{cases} 0 & h_R(t) = 0 \\ e_1(t) & h_R(t) > 0 \end{cases}, \quad \dot{x}_{I2}(t) = \begin{cases} 0 & h_R(t) = 0 \\ e_2(t) & h_R(t) > 0. \end{cases} \tag{26}$$

The temperature control could also be switched on at the beginning of the start-up process. Then the reboiler could be preheated. However, the reboiler temperature could then achieve the set point, before liquid arrives at the reboiler.

The second switching procedure occurs at the beginning of stage (II), because the dynamic behavior of the column is changing when vapor is rising. In the strict sense, the controller parameters are changing when boiling conditions have been achieved in the reboiler:

$$\begin{aligned} &K_{P1} = K_{P1}^{(I)}, K_{I1} = K_{I1}^{(I)}, K_{P2} = K_{P2}^{(I)}, K_{I2} = K_{I2}^{(I)} \qquad p_{bub,R} \le p_0 \ \ (p_R = p_0) \\ &K_{P1} = K_{P1}^{(II)}, K_{I1} = K_{I1}^{(II)}, K_{P2} = K_{P2}^{(II)}, K_{I2} = K_{I2}^{(II)} \quad p_{bub,R} > p_0 \ \ (p_R > p_0). \end{aligned} \tag{27}$$

Additionally, an anti-reset windup (ARW) strategy is combined with the PI controllers. This action is necessary if the calculated input variables u do not hold the following constraints: $0 \le u_1(t) \le F_{max}$ and $0 \le u_2(t) \le Q_{R,max}$. In such situations the derivatives of the controller state variables are set to zero: $\dot{x}_{I1}(t) = 0$ and $\dot{x}_{I2}(t) = 0$. Obviously, increasing the maximum values of the reboiler heat duty $Q_{R,max}$ and the feed flow rate F_{max} allows an acceleration of the fill-up and heating process.

The control strategy is combined with recycling partial bottom product back to the feed (Fig. 5). This implies several advantages. Such a recycle can reduce the waste of product during start-up. The trays above the reboiler are heated before vapor rises. The bottom product recycling should be applied if the discontinuous stage is accelerated and no complications occur. The bottom flow rate $B(t)$ is split into two parts:

$$B(t) = B_F(t) + B_{out}(t), \tag{28}$$

where $B_F(t)$ characterizes the flow which is added to the feed, and $B_{out}(t)$ characterizes the flow which is withdrawn from the system. The split factor

$$\kappa = \frac{B_F(t)}{B_{out}(t)} \tag{29}$$

is defined as the ratio of the recycled to the withdrawn stream. A split factor $\kappa = 10$ means, that 10 times as much is recycled to the feed as is withdrawn from the column. From (28) and (29) we obtain:

$$B_{out}(t) = \frac{1}{1+\kappa} B(t), \quad B_F(t) = \frac{\kappa}{1+\kappa} B(t). \tag{30}$$

When applying bottom product recycling, the balance equations have to be adapted accordingly.

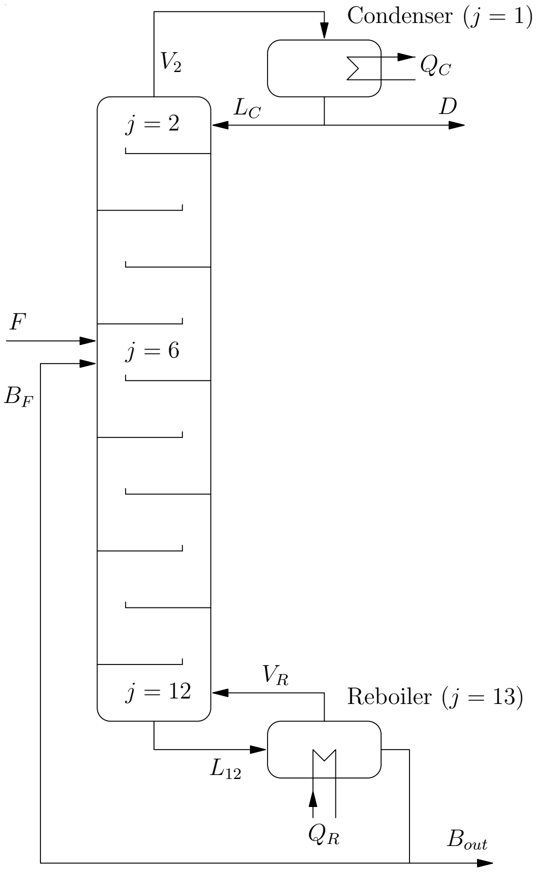

Figure 5: Ethyl acetate reactive distillation column, including bottom product recycling.

4. Process Description

To test and to demonstrate the proposed control strategy, the RD process of esterification of ethanol with acetic acid to form ethyl acetate and water is investigated. This system has also been investigated in other publications, for instance Lee and Dudukovic, 1998; Kenig et al., 2001; Alejski and Duprat, 1996 and particularly with regard to start-up Reepmeyer et al., 2003, 2004a, 2004b; Reepmeyer, 2004.

The reactive distillation plant is shown in Fig. 1. Technical data have been taken from Lee and Dudukovic, 1998 and Reepmeyer, 2004. The column consists of 13 trays ($nt = 13$). Tray 1 is the condenser, tray 13 is the reboiler, and tray 6 is the feed tray. The reaction is described by

$$\underset{(i=1)}{C_2H_5OH} + \underset{(i=2)}{C_2H_3OOH} \leftrightarrow \underset{(i=3)}{C_2H_2OOC_2H_5} + \underset{(i=4)}{H_2O}, \tag{31}$$

which takes place in every tray. The kinetics of the uncatalysed reaction is modeled by Arnikar et al., 1970:

$$r(t) = 0.485 e^{-\frac{59871.24}{RT(t)}} C_1(t)C_2(t) - 0.123 e^{-\frac{59871.24}{RT(t)}} C_3(t)C_4(t), \quad C_i(t) = \frac{x_i(t)}{v_i^L}. \tag{32}$$

The process configurations are summarized in Table 1. The physical properties to

Parameter	**Value**	**Parameter**	**Value**
nt	13	feed tray	6
d_{col}	$0.6m$	L_{weir}	$0.457m$
H_{weir}	$0.05m$	tray height	$0.34m$
jacket thickness	$0.004m$	A_{holes}	$0.0145m^2$
ν	$[-1,-1,1,1]$	R	$8.3145 J/K/mol$
v_1^L (ethanol)	$58.04 \cdot 10^{-6} m^3/mol$	v_2^L (acetic acid)	$57.24 \cdot 10^{-6} m^3/mol$
v_3^L (ethyl acetate)	$97.94 \cdot 10^{-6} m^3/mol$	v_4^L (water)	$18.02 \cdot 10^{-6} m^3/mol$
$h_{R,sp}$	$0.035m$	$T_{R,SP}$	$366K$
x_1^F (ethanol)	0.4808	x_2^F (acetic acid)	0.4962
x_3^F (ethyl acetate)	0	x_4^F (water)	0.0229
m_j	$30kg$	m_R	$30kg$
$c_{p,j}$, $c_{p,R}$ (steel)	$490 J/kg/K$	T_0, T^F	$298.15K$
$Q_{R,max}$	$80.6 \cdot 10^3 J/s$	p_0	$10^5 Pa$
F_{max}	$1.076 mol/s$	B_0	$0.868 mol/s$
c_w	2.463		

Table1: Process parameters for esterification of ethanol with acetic acid

calculate the molar enthalpies $H_{i,j}^L(t)$, $H_{i,j}^V(t)$, and the molar volume $v_{i,j}^V(t)$ respectively the density $\rho_{i,j}^V(t)$ of the pure components are adopted from the gPROMS ideal physical properties foreign object (IPPFO) database (Ideal physical properties foreign object, 2004). The vapor pressure $p^V(t)$ is computed by the Riedel-equation:

$$\ln p^V(t) = \alpha_1 + \frac{\alpha_2}{T(t)} + \alpha_3 \ln T(t) + \alpha_4 T(t)^{\alpha_5}, \tag{33}$$

because the thermodynamic data $\alpha_1, \ldots, \alpha_5$ (Table 2, taken from Perry and Green, 1997) are valid for a wide temperature range, which is needed for the start-up process.

	α_1	α_2	α_3	α_4	α_5
Ethanol ($i = 1$)	74.475	-7164.3	-7.327	$3.134 \cdot 10^{-6}$	2
Acetic acid ($i = 2$)	53.27	-6304.5	-4.2985	$8.8865 \cdot 10^{-18}$	6
Ethyl acetate ($i = 3$)	66.824	-6227.6	-6.41	$1.7914 \cdot 10^{-17}$	6
Water ($i = 4$)	73.649	-7258.2	-7.3037	$4.1653 \cdot 10^{-6}$	2

Table 2: Riedel-coefficients

The activity coefficient $\gamma_i(t)$ is calculated by the Wilson-model:

$$\ln \gamma_i(t) = 1 - \ln\left(\sum_{k=1}^{nc} x_k(t) \Lambda_{i,k}(t) \right) - \sum_{l=1}^{nc} \frac{x_l(t) \Lambda_{l,i}(t)}{\sum_{n=1}^{nc} x_n(t) \Lambda_{l,n}(t)}, \tag{34}$$

with:

$$\Lambda_{i,k}(t) = \frac{v_k^L}{v_i^L} e^{-\frac{\lambda_{i,k}}{T(t)}}. \tag{35}$$

This approach is recommended for this process (Reepmeyer, 2004). It is relatively simple from the mathematical point of view, and easy to implement. The physical constants $\lambda_{i,k}$ (Table 3) have been taken from Barbosa and Doherty, 1988. The orifice coefficient c_w has been calculated with a simplification for sieve trays with thin plates and sharp edged holes (Stichlmair, 1998):

$$c_w = \left(\frac{1}{\beta} - \Phi \right)^2, \tag{36}$$

with the discharge coefficient $\beta = \pi/(\pi + 2) = 0.611$ and the relative free area $\Phi = A_{holes}/A_{tray} = 0.0145/0.2157 = 0.0672$.

($\lambda_{i,k}$)	Ethanol ($\lambda_{i,1}$)	Acetic acid ($\lambda_{i,2}$)	Ethyl acetate ($\lambda_{i,3}$)	Water ($\lambda_{i,4}$)
Ethanol ($\lambda_{1,k}$)	0	101.6588	288.2011	198.1757
Acetic acid ($\lambda_{2,k}$)	-130.6527	0	1749.9343	2.031
Ethyl acetate ($\lambda_{3,k}$)	28.8790	-464.1592	0	26981.1421
Water ($\lambda_{4,k}$)	466.1059	403.1564	1195.67	0

Table 3: Wilson-coefficients

5. Controller Design and Simulation

The proposed strategy is applied to the ethyl acetate reactive distillation column. After fill-up and heating the reboiler temperature and the liquid level should achieve the following values $T_R = 366K$ and $h_R = 0.035m$ (Lee and Dudukovic, 1998; Reepmeyer, 2004) and should be kept constant during stage (II). Because of numerical reasons two assumptions have to be made. The trays are contaminated with the feed liquid: $x_{i,j}(0) = x_i^F$, $h_j(0) = h_R(0) = 10^{-6} m$. When the liquid is arrived at the reboiler the bottom flow rate B_0 is added as a step response of a PT_1 element instead as a step function.

The control design strategy is as follows. At the beginning, the control device for stage (I) is taken into account. The synthesis and the simulation of the control loop are realized only with the model for stage (I). No vapor is considered. First, the level control loop is designed. The temperature control unit is designed in a subsequent step. The controller parameters are chosen such that both process variables $h_R(t)$ and $T_R(t)$ achieve the set points simultaneously. For this example faster reboiler temperature dynamics are possible (Sommer et al., 2006). Then, boiling conditions will be achieved in the reboiler ($p_{bub,R} > p_0$) before the liquid level $h_R(t)$ reaches its set point. With other words, the switching procedure from stage (I) into stage (II) takes place before the fill-up process is finished completely. The controller settings are:

$$\begin{aligned} K_{P1}^{(I)} &= 200, & K_{I1}^{(I)} &= 2, \\ K_{P2}^{(I)} &= 1000, & K_{I2}^{(I)} &= 3.33. \end{aligned}$$

Now, stage (II) is considered. After vapor is rising from the reboiler, the temperature dynamics are decelerated. The temperature control has to be adjusted. Strictly speaking, the gains K_{P2} and K_{I2} are increased. The controller settings of the level control unit are retained. We obtain:

$$K_{P1}^{(II)} = 200, \quad K_{I1}^{(II)} = 2,$$
$$K_{P2}^{(II)} = 2000, \quad K_{I2}^{(II)} = 6.67.$$

The simulation of the discontinuous stage stops, if vapor arrives at the condenser. The results are presented in Fig. 6 and Fig. 7. The discontinuous stage is finished after 4292s. The liquid level $h_R(t)$ and the temperature $T_R(t)$ reach the set points with steady state accuracy and no overshoot occurs. All in all, promising simulation results are obtained. Two single PI control loops are adequate. A multivariable PI controller could not provide better results. The mole fraction profile is presented in Fig. 8. Here, the mole fraction values of all components at the end of the discontinuous stage are illustrated as a function of the column trays.

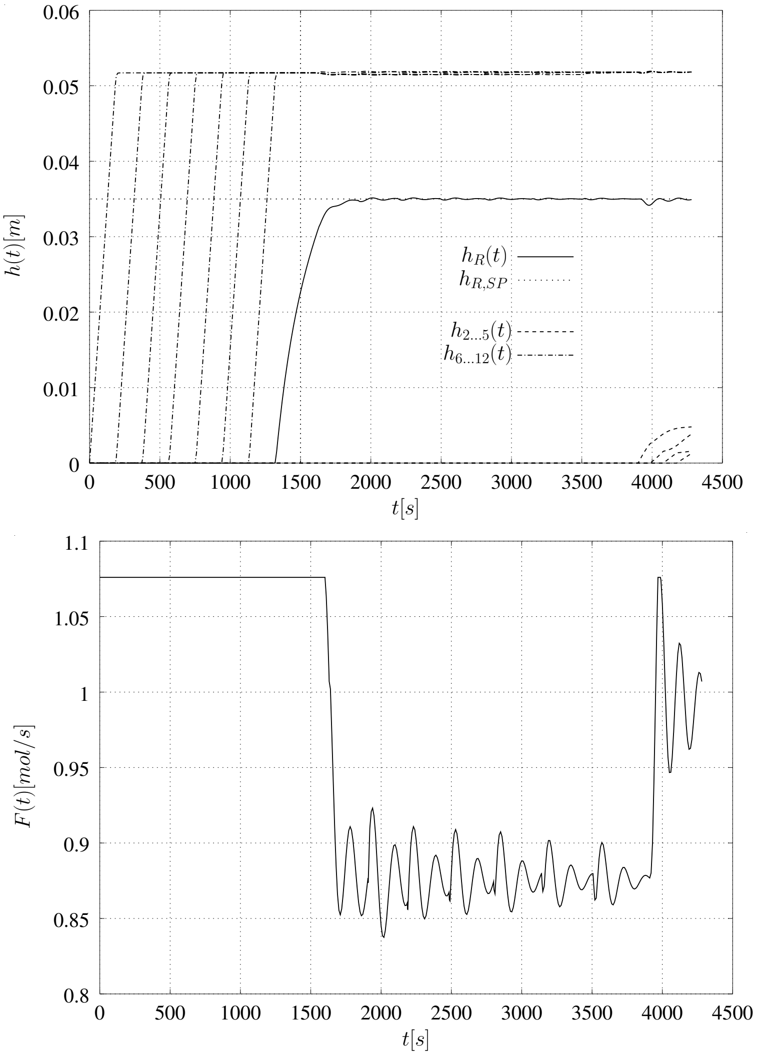

Figure 6: Reboiler liquid level control of the discontinuous stage.

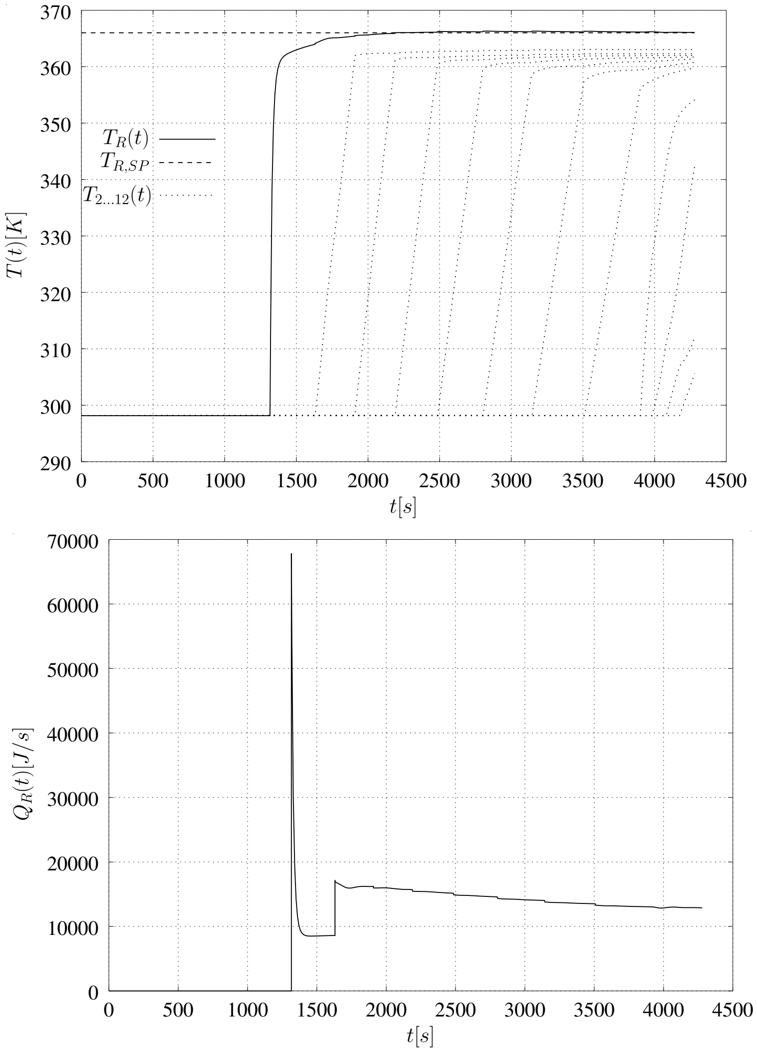

Figure 7: Reboiler temperature control of the discontinuous stage.

Now, we include bottom product recycling. The split factor $\kappa = 3$ is used. The controller settings for stage (I) are retained and the parameters for stage (II) are changed:

$$K_{P1}^{(I)} = 200, \quad K_{I1}^{(I)} = 2,$$
$$K_{P2}^{(I)} = 1000, \quad K_{I2}^{(I)} = 3.33,$$

$$K_{P1}^{(II)} = 150, \quad K_{I1}^{(II)} = 1.36,$$
$$K_{P2}^{(II)} = 2000, \quad K_{I2}^{(II)} = 23.53.$$

After the liquid has been arrived at the reboiler, an amount of $B_F = 0.651 mol/s$ will be added to the feed flow rate $F(t)$ and a value of $B_{out} = 0.217 mol/s$ will be withdrawn from the column. Thus, 75% of the bottom flow rate is recycled back to the feed. The simulation results are demonstrated in Fig. 9 and Fig. 10.

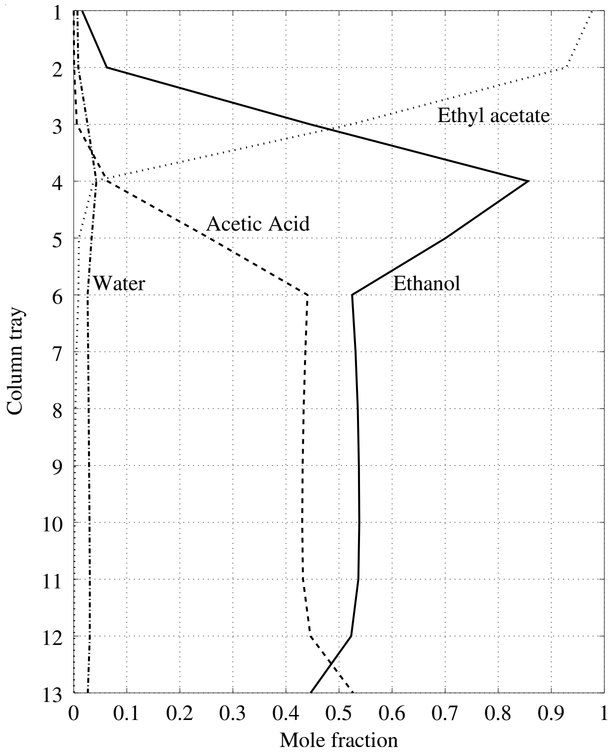

Figure 8: Mole fraction profile of the discontinuous stage.

With recycling, the discontinuous stage takes $2695s$. Compared with no recycling, the discontinuous stage is accelerated. However, we obtain an overshoot for both process variables. The trays above the reboiler are heated, while no vapor rises. The flow rate B_F can be seen as a disturbance of the control loop at the plant input. This disturbance is compensated by both control units such that steady state accuracy of the process variables liquid level $h_R(t)$ and temperature $T_R(t)$ can be achieved. The mole fraction profile is shown in Fig. 11.

The bottom product recycling is recommended for start-up the discontinuous stage of the ethyl acetate RDC, because the dynamic behavior does not change significantly, and the fill-up process will be accelerated.

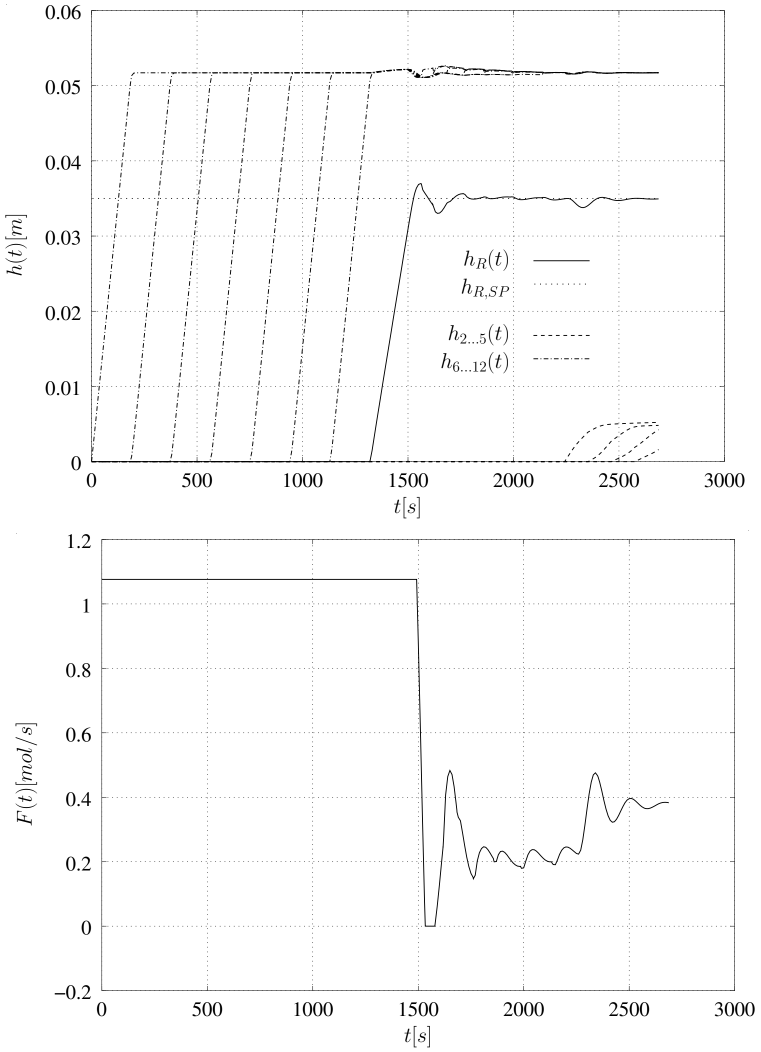

Figure 9: Reboiler liquid level control of the discontinuous stage including bottom product recycling.

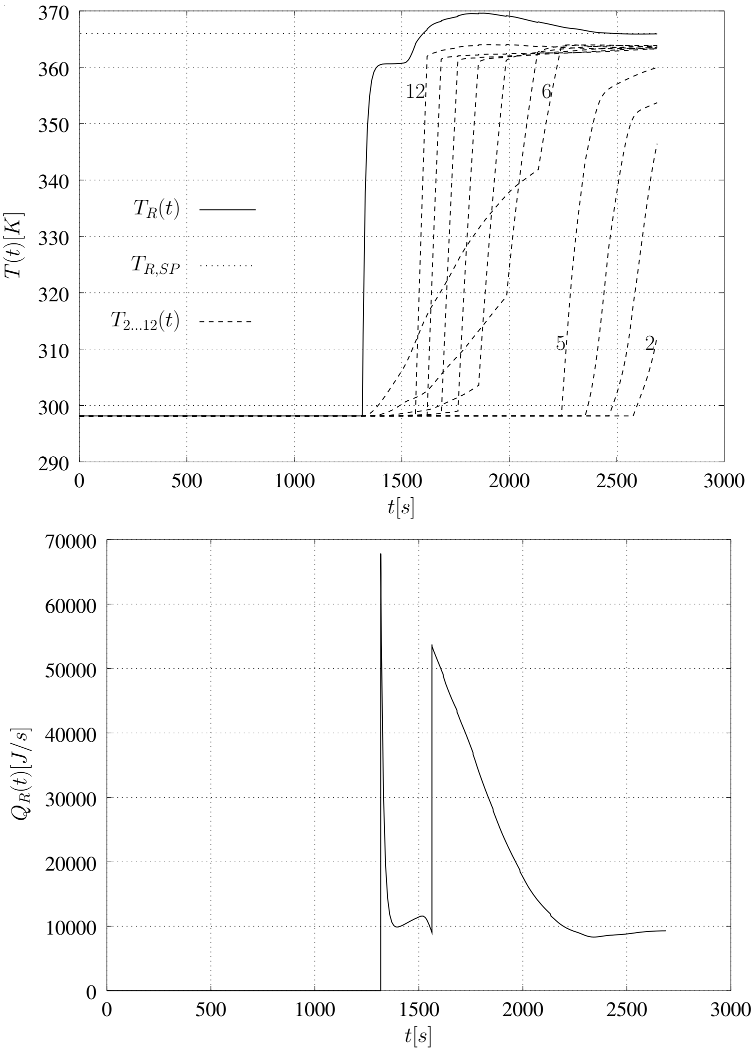

Figure 10: Reboiler temperature control of the discontinuous stage including bottom product recycling.

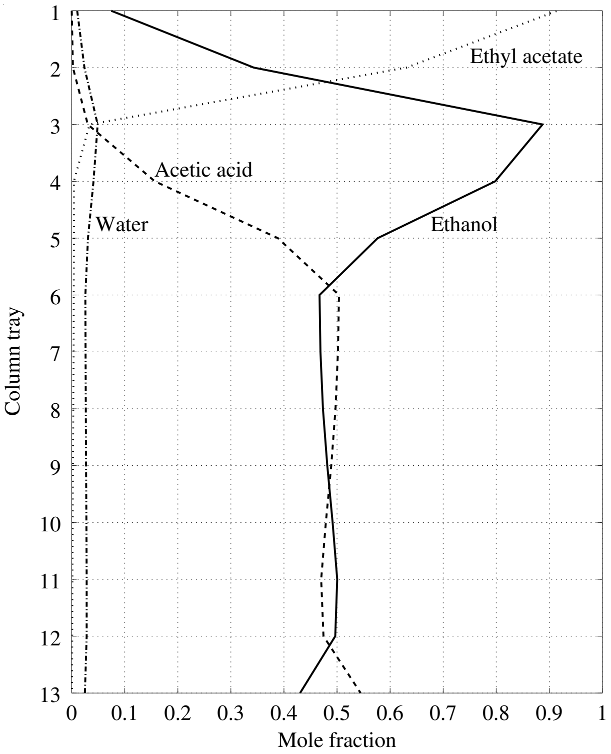

Figure 11: Mole fraction profile of the discontinuous stage including bottom product recycling.

6. Conclusion

In this paper we propose a feedback concept for start-up of empty cold reactive distillation columns. To solve the control problem the start-up process has been divided into four stages. We consider stages (I) and (II), i.e. the discontinuous stage of start-up, and a corresponding model has been developed.

Our control strategy can be summarized as follows. The feed flow rate $F(t)$ is used to control the reboiler liquid level $h_R(t)$ and the reboiler heat duty $Q_R(t)$ is used to control the reboiler temperature $T_R(t)$. The bottom flow rate is set to its steady state value: $B(t) = B_0$. Two single control loops with PI controllers, including anti-reset windup, are adequate. During the automatic start-up of the discontinuous stage two switching procedures are necessary. The control concept is combined with bottom product recycling. Thereby, only a small part of the bottom product has to be disposed.

The start-up strategy is tested for the esterification of ethanol with acetic acid. Promising simulation results are obtained. The approach with bottom product recycling is recommended for the presented discontinuous stage.

In a subsequent step, control policies have to be developed for the next start-up stages to formulate a complete start-up strategy based on feedback control. The combination of the mentioned start-up approach with bottom and top product recycling as well as initial charge of products and educts will also be investigated.

7. Nomenclature

A	area, m^2
A_j	active area of a tray, m^2
A_{holes}	total hole area, m^2
$B(t)$	bottom flow rate, mol/s
c_p	specific heat capacity, $J/kg/K$
c_w	orifice coefficient
$C(t)$	concentration, mol/m^3
$D(t)$	distillate flow rate, mol/s
d	diameter, m
$e(t)$	control error
$E(t)$	energy, J
$F(t)$	feed flow rate, mol/s
$h(t)$	liquid level, m
$H(t)$	molar enthalpy, J/mol
H_{weir}	weir height, m
K_P, K_I	controller parameters
$L(t)$	liquid flow rate, mol/s
L_{weir}	weir length, m
m	mass, kg
$M(t)$	liquid molar hold-up, mol
MW	molar weight, kg/mol
nc, nt	number of components, trays
$p(t)$	pressure, Pa
$Q(t)$	heat duty, J/s
r	radius, m
$r(t)$	reaction rate, $mol/m^3/s$
R	ideal gas constant, $J/mol/K$
$T(t)$	temperature, K
$u(t)$	controller output, manipulated variable
$v(t)$	molar volume, m^3/mol
$V(t)$	vapor flow rate, mol/s
$x(t)$, $y(t)$	liquid mole fraction, vapor mole fraction
$x^{mass}(t)$, $y^{mass}(t)$	liquid mass fraction, vapor mass fraction
$x_{I1}(t)$, $x_{I2}(t)$	controller state variables

Greek letters:

$\alpha_{1..5}$	Riedel coefficients
β	discharge coefficient
$\gamma(t)$	activity coefficient
$\varepsilon(t)$	reaction volume, m^3
θ	angle, *rad*
κ	split factor
λ	Wilson-coefficient
ν	stoichiometric coefficient
$\rho(t)$	density, kg/m^3
Φ	relative free area

Subscript:

C	condenser
col	column
down	down comer
F	feed
i, j	component, tray
max	maximum value
R	reboiler
SP	set point
0	steady state

Superscript:

F	feed
L	liquid
V	vapor

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