# Evaluation of Dynamic Models of Distillation Columns with Emphasis on the Initial Response

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**Abstract** A rigorous model for staged high purity distillation based on first principles is developped. The importance of the tray hydraulics to obtain good agreement between simulation and experiment is demonstrated. Analytical expressions to derive the hydraulic time constants $\tau_l$, $\tau_v$ and the vapor constant for models with simplified tray hydraulics are presented. The validity of these key parameters is verified by comparison to experimental data.

**Key Words** Distillation dynamics; Initial time constant

# 1 Introduction

Simplified models which simplify or neglect tray hydraulics and energy balance are often used for studies of distillation column dynamics and control. However, the applicability of such simple models for this purpose is often questioned by practioneers. This critique is indeed reasonable as one knows that the tray hydraulics are crucial in determining the initial dynamic response, which is of key importance for control.

The objective of the paper is to derive models that are as simple as possible while at the same time match the behavior of real columns. We consider two kinds of models: 1) Simplified models where the tray hydraulics is taken care by simple parameters such as the hydraulic time constant $\tau_L$ and the vapor constant $\lambda$; and 2) Models with detailed description of the tray hydraulics.

The most important parameters from an operational point of view are the liquid holdup $M_l$, the hydraulic time constant $\tau_l$, the parameter $\lambda$ for the initial effect of a change in vapor flow on liquid flow, the fraction of vapor on the tray and the pressure drop. These key parameters may be determined from either 1) experiments, 2) simulation or 3) estimated from analytical expressions based on linearizing the detailed model equations. One problem is to get consistent results from the different methods.

In this paper we compare the dynamics of a laboratory-scale high purity distillation column with those of a rigorous and simplified dynamic process model. We find that a very detailed model is needed to match closely the responses of the real experimental column. The objective of the experimental work was to evaluate the theoretical simulation results and gain further insight into the complexity of the required model.

# 2 Modelling

The rigorous model has a seperate mass and energy balance for holdups on tray and downcomer. The holdup on the stage is computed from pressure drop correlations and the geometry. The downcomer is modelled as a mixing tank. The flash calculation is performed as a UV-flash.

For the second model the molar liquid holdup of the stage is the combined holdup of tray and downcomer, the vapor holdup is neglected. The tray hydraulics is linearized with one hydraulic time constant for the liquid flow and a vapor constant to describe the vapor hydraulics. Since the flash calculation is reduced to a p,x-flash, pressure is not longer the driving force of the system. The liquid flow from the stage is computed by a linear function, while a steady state energy balance is applied to determine the vapor flow from the stage. The liquid time constant $\tau_L$ and the vapor constant $\lambda$ are constant for each section. To allow the thermodynamic package to compute liquid enthalpy, equilibrium constant and temperature, the pressure on each stage has to be set.

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## 2.1 Rigorous Tray Model

**Assumptions** The total holdup of the tray consists of liquid and vapor holdup. According to the geometry of the interiour of the distillation column, see Fig.: 1, the liquid holdup on the tray is devided into liquid on the active tray area and liquid in the downcomer. The following assumptions are made:

- R1 two-phase system in thermal and mechanical equilibrium
- R2 perfect mixing in vapor and liquid phases
- R3 no heat losses to the surroundings
- R4 no heat of mixing
- R5 temperature dynamics of the column structure is neglected, this is, no heat is stored in the metal

Conservation of internal energy and constant volume of the system implies that the flash calculation is solved as an UV-flash. The assumption of identical pressure and temperature in liquid and vapor phase is necessary to enable a consistent solution of the physical properties for each component.

**Material and Energy Balance** The balances presented are valid for a stage without feed, side draw and external heating or cooling. Some modifications are made for the reboiler and downcomer.

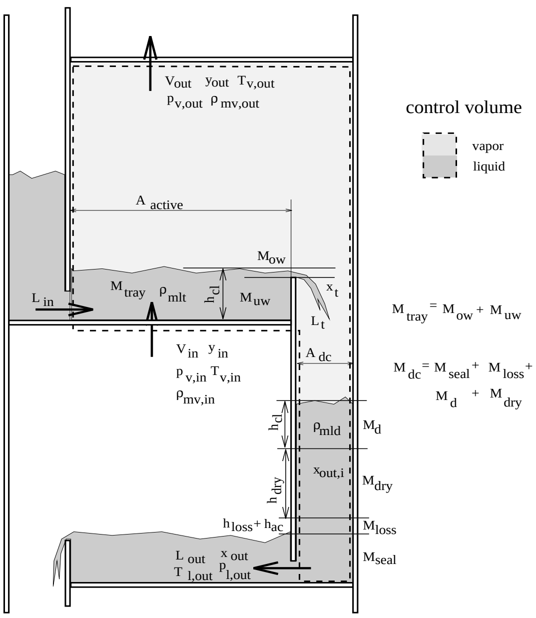


Figure 1: Control volume on a stage

$$\frac{dM_i}{dt} = V_{in}\ y_{in,i} + L_{in}\ x_{in,i} - V_{out}\ y_{out,i} - L_t\ x_{t,i} \quad (1)$$

The total component holdup on the tray, this is liquid on the tray and vapor above tray and downcomer is:

$$M_i = M_{tray}\ \ x_{t,i} + M_v\ \ y_{out,i} \quad (2)$$

The total molar holdup on the tray given by:

$$\sum_{i=1}^{n} M = M_{tray} + M_v \quad (3)$$

The energy balance of the tray

$$\frac{dU_{tray}}{dt} = V_{in} h_{v,in} + L_{in} h_{l,in} - V_{out} h_{v,out} - L_t h_{l,t} \quad (4)$$

where the total internal energy is described by

$$U_{tray} = M_v\ (h_{v,out} - \frac{p}{\rho_v}) + M_l\ (h_{l,out} - \frac{p}{\rho_l}) \quad (5)$$

A similar set of equations is applied to the downcomer material balance:

$$\frac{M_{dc,i}}{dt} = L_t\ x_{t,i} - L_{out,i}\ x_{out,i} \quad (6)$$

the component balance:

$$M_d = \sum_{i=1}^{n} M_{dc,i} \ \ ; \ \ M_{dc,i} = M_d\ x_{out} \quad (7)$$

the energy balance of the tray

$$\frac{U_{dc}}{dt} = L_t\ \ h_{l,t} - L_{out}\ h_{l,out} \quad (8)$$

and the total internal energy, were we neglect the pressure dependent part

$$U_{dc} = M_d\ h_{l,out} \quad (9)$$

Note that there is no flash calculation performed on the downcomer holdup. The vapor composition is in equilibrium with the liquid on the tray and the vapor volume of the tray and downcomer are combined.

**Vapor-Liquid Equilibrium** The thermodynamic equilibrium of vapor and liquid, is described by Raoult's law.

$$y_{out,i} = K_i\ x_{out,i} \ \ ; \ \ \sum_{i=1}^{n} y_{out} = \sum_{i=1}^{n} x_{out} \quad (10)$$

The composition of the vapor leaving the tray is computed by the Murphree tray efficiency coefficient

$$\eta_i = (y_{out,i} - y_{in,i})/(y_{eq,i} - y_{in,i}) \quad (11)$$

with $y_{eq,i}$ as the equilibrium vapor composition at given tray temperature and pressure. The composition of the vapor that would be in equilibrium, $y_{eq,i}$, with the liquid leaving the tray.

**Holdup Distribution** The liquid and vapor molar holdup on the tray is related to the total tray volume by:

$$V_{tray} = M_{tray}/\rho_l + M_v/\rho_v + M_{dc}/\rho_l \tag{12}$$

The molar volume on tray is computed by

$$M_{tray} = (h_{cl}\ A_{active}\ \rho_{ml,t})/MW_{l,t} \tag{13}$$

and of the downcomer

$$M_{dc} = (h_{dc}\ A_{dc} + V_{dc0})\ (\rho_{ml,d}/MW_{l,d}) \tag{14}$$

**Froth Density** We have choosen to use the correlation presented by Bennett (1983) to compute the froth density and thus the liquid holdup on the tray. The velocity of the vapor through the active tray area is:

$$v_{active} = (V_{in}\ MW_v)/(A_{active}\ \rho_{mv}) \tag{15}$$

and the free (hole) area of the sieve tray.

$$v_{hole} = v_{active}\ A_{active}/A_{hole} \tag{16}$$

the dimensionless (superficial) velocity factor

$$K_s = v_{active}\sqrt{\rho_{mv}/(\rho_{ml} - \rho_{mv})} \tag{17}$$

the froth density $\phi$ is expressed by

$$\phi = exp(\pi_1\ K_s^{\pi_2}) \tag{18}$$

with these parameters the clear liquid height $h_{cl}$ on the tray is

$$h_{cl} = \phi\ (h_{weir} + (h_{ow}/\phi)^{\pi_3}) \tag{19}$$

The parameter $\pi_1$ to $\pi_3$ are empirical constants determined from experiments. If the computed froth height, $h_{cl}/\phi \leq h_{weir}$, the liquid leaving the tray is set to zero.

**Hydraulic Model** The modeling of the tray hydraulic is based on emperical correlation selected from the literature. These correlations are not necessarily the best available. The applied correlations allow fairly accurate predictions and are easy to implementation in the model.

**Flow over outlet weir** The liquid flowing over the circular weir from the tray to the downcomer, is computed with a modified Francis weir formula (Perry *et.al.*, 1984). Since the outlet weir is placed off center towards the column wall, the liquid height above the weir $h_{ow}$ will not be constant. There are no existing correlations which deal with the converging flow over outlet weirs in distillation column (Lockett, 1986). Taking the design into consideration, we choose to correct the weir length with a factor of 0.5.

$$h_{ow} = 44300(q/(0.5\ \ d_{weir}))^{0.704} \tag{20}$$

**Downcomer** The pressure drop over the downcomer (Perry *et. al* 1984) from the surface of the liquid exiting the downcomer to the surface of the downcomer level is identical to the total pressure drop over a plate. The flow under the downcomer apron is modelled by an instationary Bernoulli equation. The computation takes the pressure loss due to friction and the acceleration of the liquid under the downcomer apron into consideration.

$$dp_{plate}/\rho_{ml} = g\ h_{dc} - \zeta\ v_{dc}^2 - L\ \ v_{dc}/dt \tag{21}$$

where the velocity under the apron is

$$v_{dc} = q_t/A_apron \tag{22}$$

and the characteristic length L is choosen to be equal to the height of the downcomer apron.

**Pressure drop correlation** The total pressure drop $dp_{plate}$ over a plate is the difference of the pressure of the vapor entering the tray and the vapor leaving the tray:

$$dp_{plate} = p_{v,in} - p_{v,out} = dp_{static} + dp_{dry} \tag{23}$$

This is equal to the sum of the liquid head on the tray, $dp_{static}$ and the dry pressure drop $dp_{dry}$. The pressure drop due to the liquid head on the tray is

$$dp_{static} = g\ h_{cl}\ \rho_{ml} \tag{24}$$

According to Lockett (1986) the hydraulic gradient for sieve tray distillation column of small diameter (less than 0.5 m) is negligible. Numerous correlations for the dry pressure drop are available (*e.g.*: Liebson *et. al.* (1957))we have choosen:

$$dp_{dry} = \rho_{ml}\frac{51}{C_o^2}\frac{\rho_{mv}}{\rho_{ml}}v_{hole}^2 \tag{25}$$

**Thermodynamic Properties** The thermodynamic properties of the components are calculated by the ASPEN PROPERTIES PLUS (1988) package.

# 3 Simplified Model with Linearized Tray Hydraulics

Modern control system synthesis is based on the state variable representation of the plant model. In applying these tools to higher order systems, such as distillation column, practical reasons during controller design require an order reduction. This is the large number of state variables obtained by models based on first principle has to be replaced by a considerably smaller set of a suitable set of state variables. The prefered model is obvious one with a first order plus dead time characteristic for the stage model. During model reduction the hydraulic lag (of the liquid flow) has to be preserved. Models with linearized tray hydraulics are most common in practical use (Rademaker (1975), Skogestad (1988)) as this simplifies the model considerably.

## 3.1 Tray Model

**Model** The holdup of tray and downcomer are combined to one liquid holdup which is similar to the total liquid holdup of the rigorous model. The linearization of the tray hydraulics (Eq. 27) is done based on the hydraulic relationships of the rigorous model (see Eq. 12 to 25). The energy balance is done over the entire stage holdup. Additionally to the assumption R1 to R5 for the rigourous model the following assumptions are made:

SH1 vapor holdup neglected

SH2 combine tray and downcomer to one liquid holdup

SH3 assume $u_l \approx h_l$ , $\frac{dh_{l,out}}{dt} \approx 0$ , this is a rather rough assumption, especially since the reference state for the thermodynamic is $25^oC$

SH4 linearized tray hydraulics

SH5 $p =$ const on each tray

Under consideration of assumption SH1 to SH2 the material balance is similar to Eq. 1 to Eq. 3. Assumption SH3 simplifies the energy balance, which is now used to compute the vapor flow leaving the tray:

$$V_{out} = \frac{1}{h_{v,out} - h_{l,out}} (V_{in}\ (h_{v,in} - h_{l,out}) + L_{in}\ (h_{l,in} - h_{l,out})) \qquad (26)$$

The vapor-liquid equilibrium is described by Eq. 10. The tray hydraulics is expressed by:

$$L_{out} = L_{out,0} + \lambda(V_{in} - V_{in,0}) + \frac{1}{\tau_l}(M_l - M_{l,0}) \quad (27)$$

## 3.2 Linearized Tray Hydraulics

In this section we use the simplified tray hydraulics which is proposed by Rademaker (1975) and Skogestad (1988). Simple expressions for the key parameters describing the hydraulics, namley the hydraulic time constant $\tau_l$ and the effect of vapor flow on liquid flow the vapor constant $\lambda$ will be derived. For simplicity, the vapor holdup is neglected and constant molar flow is assumed $V_{in} = V_{out}$.

### 3.2.1 Linearization

It is assumed that the liquid flow, $L_{out}$, from a stage is directly affected through the vapor flow in ,$V_{in}$, (indirectly through its effect on the liquid level) and by the holdup (mass) on the tray $M$. Linearizing this relation yields (Rademaker *et.al.*(1975), Skogestad and Morari (1988))

$$\begin{aligned} dL_{out} &= \left(\frac{\delta L_{out}}{\delta V_{in}}\right)_M dV_{in} + \left(\frac{\delta L_{out}}{\delta M}\right)_V dM \\ &= \lambda\ dV_{in} + \frac{1}{\tau_L}\ dM \end{aligned} \qquad (28)$$

Note that this relationship is assumed to hold dynamically. We want to obtain $\lambda$ and $\tau_l$ from steady state relationships for the liquid holdup on the stage found from design data of the column. The liquid holdup can be expressed by the following relations $M = f(L, V, dp)$ with pressure drop $dp = f(L, V)$. Linearize these expressions and assume $L_{in} = L_{out}$ yields:

$$\begin{aligned} dM &= \left(\frac{\delta M}{\delta L_{out}}\right)_V dL_{out} + \left(\frac{\delta M}{\delta V_{in}}\right)_L dV_{in} \\ &= \tau_L\ dL_{out} + \tau_V\ dV_{in} \end{aligned} \qquad (29)$$

Note that this equations only holds at steady state. After obtaining $\tau_l$ and $\tau_v$ we may express $\lambda$ by

$$\lambda = -(\tau_V / \tau_L) \qquad (30)$$

This follows by setting $dM = 0$ in Eq. 28 and 29 and rearrange to get $(\delta L_{out}/\delta V_{in})_M$.

### 3.2.2 Estimation of hydraulic time constants from column design data

The liquid holdup distrubution on the stage (tray plus downcomer) is shown in Fig.: 1. To simplify the derivation the vapor holdup is neglected and all liquid holdup is in terms of clear liquid. The liquid holdup depends on geometry, liquid and vapor flow. The liquid holdup of a stage can be split up into liquid on the tray $M_{tray}$ and in the downcomer $M_d$. The liquid holdup on the tray can be divided into liquid under, $M_{uw}$, and over, $M_{ow}$, weir. The holdup in the downcomer consists of the liquid in the seal pan $M_{seal}$ (or behind the inlet weir), holdup due to hydrodynamic losses under the downcomer apron, $M_{loss}$, holdup due to dry pressure drop $M_{dry}$ and the amount of liquid corresponding to the clear liquid height on the tray $M_{dc}$. The liquid holdup in the seal pan is independent of the vapor or liquid flow. Since the height of clear liquid on the tray and the corresponding height of liquid in the downcomer (denoted

$h_{cl}$ in Fig.:1 ) depend equally on liquid and vapor flow, these holdups are combined to $M$. To simplify the computation of the partial derivatives we combine the liquid holdups on the stage dependent on the vapor flow to $M_{cl}$ and consider the liquid flow dependent part $M_{ow}$ separately.

The holdup is related to geometry and flows by the following relations:

$$M = f(L_{out}, V_{in}) \quad ; M = M_{tray} + M_{dc} \tag{31}$$

$$M_{tray} = M_{uw} + M_{ow} = A_{active}\, \rho_l\, h_{cl} \tag{32}$$

$$M_{dc} = A_{dc}\, \rho_l\, h_{cl} \tag{33}$$

$$M_{ow} = f(L_{out}) \;\; ; M_{ow} = A_{active} \rho_l\, h_{ow} \tag{34}$$

$$M_{dry} = f(V_{in}) \;\; ; M_{dry} = A_{dc}\, dp_{dry}/(g\; MW_l) \tag{35}$$

$$M_{loss} = f(L_{out}) \;\; ; M_{loss} = A_{dc}\, \rho_l\, (\zeta\, v_{dc}^2/g) \tag{36}$$

The clear liquid height is defined in Eq. 19. The height over weir $h_{ow}$ is obtained from Eq. 20, finally the dry pressure drop is computed from Eq.: 25.

Assuming constant molar flows over the tray we can express the hydraulic time constant of vapor $\tau_v$ and liquid $\tau_l$ of Eq.: 29 by changes in liquid holdup on the stage.

$$\tau_V = \left(\frac{\delta M_{dry}}{\delta_V}\right)_L + \left(\frac{\delta M_{cl}}{\delta_V}\right)_L \tag{37}$$

$$\tau_L = \left(\frac{\delta M_{loss}}{\delta_L}\right)_V + \left(\frac{\delta M_{ow}}{\delta_L}\right)_V \tag{38}$$

Express the holdups with correlations 32 to 36 and linearize the nonlinear equations yields:

$$\frac{\delta M_{cl}}{\delta V} = M_{cl}\, \frac{\phi(ln\phi)^2}{V_{in}} \tag{39}$$

$$\frac{\delta M_{dry}}{\delta V} = 2\, \frac{M_{dry}}{V_{in}} \tag{40}$$

$$\frac{\delta M_{loss}}{\delta L} = 2\, \frac{M_{loss}}{L_{out}} \tag{41}$$

$$\frac{\delta M_{ow}}{\delta L} = 0.704\, \frac{M_{ow}}{L_{out}} \tag{42}$$

Combining Equations 39 to 42 with 37 and 38 yields:

$$\tau_V = 2\, \frac{M_{dry}}{V_{in}} + M_{cl}\, \frac{\phi(ln\phi)^2}{V_{in}} \tag{43}$$

$$\tau_L = 2\, \frac{M_{loss}}{L_{out}} + 0.704\, \frac{M_{ow}}{L_{out}} \tag{44}$$

$$\lambda = -\frac{\tau_V}{\tau_L} = -\frac{2M_{dry} + \phi(ln\phi)^2 M_{cl}}{2M_{loss} + 0.704 M_{ow}}\, \frac{L_{out}}{V_{in}} \tag{45}$$

# 4 Obtain operational parameters from experiment

## 4.1 Liquid holdup and distribution on tray $M_l$ and $M_d$

i) From geometric data and pressure drop measurement over the column the amount of liquid stored on a stage will be the sum of liquid on the tray itself and the liquid stored in the downcomer. From the column design we know the area of the tray $A_{active}$ and downcomer $A_{dc}$ and the volume of the downcomer seal $V_{dc,0}$. For simplicity we neglect the hydrodynamic pressure drop under the downcomer apron, such that $h_{dc} = h_{tot}$. Pressure drop measurement will give the clear liquid height $h_{cl}$ on the tray and the backup in the downcomer (see Fig.: 1).

$$M_l/\rho_l \approx A_{tray}\, h_{cl} + A_{dc}\, h_{tot} + V_{dc,0} \tag{46}$$

$$h_{tot} = dp_{plate}/(\rho_{ml}\, g) \approx h_{dry} + h_{cl} \tag{47}$$

The liquid height corresponding to the dry pressure drop $h_{dry}$, $h_{dry}$ is determined from Eq.: 25 and the clear liquid height $h_{cl}$ from Eq.: 19.

ii) Measuring the initial temperature response to a step change in reflux $\Delta L$ or vapor flow $\Delta V$ enable the estimation of the liquid holdup. The initial response of the composition, $\Delta x_i$ on the tray is independent on the magnitude of the applied step change (Skogestad, 1988). From a mass balance over a stage it will be possible to estimate the tray holdup.

OP1 vapor holdup is negligible

OP2 neglect the energy balance, which results in equimolar flows, that is $L_i = L_{i-1}$ and $V_i = V_{i+1}$

OP3 constant molar liquid holdup $M$

$$dM_i/dt = L_{i-1} - L_i + V_{i+1} - V_i \tag{48}$$

$$M\; dx_i/dt = L_i\; (x_{i-1} - x_i) + V_i\; (y_{i+1} - y_i) \tag{49}$$

For a step change in $L_i$ and $V_i$ the internal flows are $L_i = L_i^o + \Delta L$ and $V_i = V_i^o + \Delta V$. Substracting the steady state solution of Eq. 48 from Eq. 49 and assuming that immediately after the step change the tray composition to be unchanged, yields a linear equation in $\Delta L$ and $\Delta V$:

$$M \frac{d\Delta x_i}{dt} = \Delta\; L_i(x_{i-1}^o - x_i^o) + \Delta\; V_i(y_{i+1}^o - y_i^o) \tag{50}$$

To facilitate the use of the installed measurements on the investigated distillation column we assume:

OP4 vapor-liquid equilibrium described by $y_i = K_i x_i$

OP5 a linear relation between liquid composition and temperature, *i.e.* $x_i = T_i k_i$ further assume that $k_i$ is constant for trays next to each other

Apply assumptions OP4 and OP5 and rearrange Eq.: 50 yields:

$$M_i \frac{d\Delta T_i}{dt} = \Delta L(T^o_{i-1} - T^o_i) + \Delta V K_i (T^o_{i+1} - T^o_i) \quad (51)$$

From Eq.: 51 it is possible to estimate the molar holdup on stage for a step change in reflux and vapor respectively.

iii) Determine the liquid volume inside the distillation column by empty the column. Liquid evaporating from the column interiour is collected in the accumulator. The combined holdup change of accumulator and reboiler is the amount of liquid stored under operation in the column.

### 4.2 Liquid hydraulic time constant $\tau_l$

The hydraulic time constant $\tau_l$ describe the dependency of the internal liquid flows $dL$ on the manipulated variables $dL_T$. Under the assumption of constant molar flows, the flow dynamics in a distillation column can be approximated by (Rademaker *et. al.*, 1975, Skogestad*et. al.*, 1988):

$$dL_N \approx \frac{1}{(1+\tau_l s)^N} dL_T + \left(1 - \frac{1}{(1+\tau_l s)^N}\right) \lambda dV \quad (52)$$

Eq. 52 is derived by repeated combination of the massbalance Eq. 48 assuming $V_{in} = V_o ut$ and the tray hydraulics Eq. 28 for each tray (Rademaker *et. al.*, 1975)) The response of the liquid flow on stage N is a cascade of first-order responses, one for each tray. Introduce the following approximation:

$$(1 + \tau_L s)^{-N} \approx e^{-N\tau_L s} = e^{-\theta s} \quad (53)$$

yields:

$$dL_N \approx e^{-\theta s}\, dL_T + (1 - e^{-\theta s})\, \lambda dV \quad (54)$$

From Eq. 54 we can estimate the hydraulic time constant from the measurement of the delay between a change in external reflux until the liquid outflow of tray $N$ changes $\theta_L \approx \tau_l N$. Determining the liquid hydraulic time constant experimentally can be done by observing the reboiler level after a step change in external reflux. A second method is the observation of the temperature response to an increase in $L_T$. The initial response will indicate the time a reflux flow change needs to propagate through the column.

### 4.3 Vapor constant $\lambda$

The vapor constant $\lambda$ represents the initial effect of a change in vapor flow on liquid flow from a stage. Experimentally we can obtain $\lambda$ by the following means.

i) Recording the temperature response for an increase in vapor flow V. In case of $\lambda \leq -0.5$ we will have an increase in tray temperature in the upper part of the column earlier than in the lower part. Earlier response of trays in the top section than in the bottom section is due to liquid pushed from the trays such that the internal reflux increase temporarily. This effect will be more extensive in the lower part of the column than in the upper part.

ii) Observation of the reboiler holdup after an increase in heat supply where the bottom flow is kept constant. Observing an inverse response indicate $\lambda \leq -0.5$.

iii) The estimation of $\lambda$ from differential pressure drop measurement under application of Eq. 37 to Eq. 45.

## 5 Results

### 5.1 Holdup estimation

For the estimation of holdup and time constants we assume that $\rho_{ml}$ and $MW_l$ is constant and choosen the conditions of 50/50 mixture of ethanol and butanol at boiling point and 1 bar, which gives $\rho_{ml} = 727.6 kg/m^3$, $MW_l = 60.1 kg/kmol$ on each tray and $\Delta h_{vap} = 41320 kJ/kmol$. The K-value in Eq. 51 is taken from a simulation of the vapor-liquid equilibrium at 1 bar at the initial steady state conditions.

Table 1 list the experimental conditions of experiment 2 and 3. The liquid volume in the distillation column is determined by dumping the distillation column after the experiment. The liquid volume determined by this method is denoted $V_{l,d}$. A second method to estimate the liquid volume, $V_{l,dp}$, in the column is to compute the amount of liquid on the trays from the differential pressure drop over the column under consideration of the downcomer holdup. The estimation of the liquid holdup (denoted $M_3$ and $M_9$) by measuring the **initial time constant** for a step in external reflux (Fig.: 4) and a step in heat supply (Fig.: 5) is done by applying Eq.: 51 with $\Delta V = 0$ and $\Delta L = 0$, respectively.

The liquid holdup estimated by the rigorous model ($M_R$) and determined experimental $M_{exp}$ agree very well. The holdup estimated from the initial time constant $M_{init}$ and from tht simplified model $M_{simp}$ show an extensive mismatch.

The holdup estimation from the initial time constant of the experiment relies on the accuracy in determination of the temperatuer derivatives. Determining the temperature derivative from *e.g.:* Fig.: 5 will give inaccurate results due to noise. In conjunction with the rough approximation of the physical properties an estimation of the holdup will be unreliable.

Table 1: Data for holdup estimation for experiment 2 and 3

| | dim. | Exp. 2 | Exp. 3 |
|---|---|---|---|
| $F$ | $ml/min$ | 250 | 250 |
| $z_F$ | | 0.54 | 0.56 |
| $d_p$ | $mbar$ | 22.3 | 26.1 |
| $Q_{t=0}$ | $kJ/s$ | 3.60 | 4.50 |
| $Q_{t\geq 0}$ | $kJ/s$ | 4.05 | 4.50 |
| $L_{t=0}$ | $ml/min$ | 250 | 350 |
| $L_{t\geq 0}$ | $ml/min$ | 250 | 380 |
| $V_{l,d}$ | $l$ | 2.94 | 3.18 |
| $V_{l,dp}$ | $l$ | 2.81 | 2.79 |
| $M_{exp}$ | $mol$ | 3.24 | 3.51 |
| $T_2$ | $^oC$ | 79.11 | 80.96 |
| $T_3$ | $^oC$ | 79.49 | 84.80 |
| $T_4$ | $^oC$ | 80.45 | 90.07 |
| $\Delta T_3/dt$ | $^oC/s$ | 0.0018 | 0.0133 |
| $M_{init,3}$ | $mol$ | 6.16 | 2.96 |
| $M_{simp,3}$ | $mol$ | 1.2 | 1.2 |
| $M_{R,3}$ | $mol$ | 3.05 | 2.80 |
| $T_8$ | $^oC$ | 90.87 | 109.79 |
| $T_9$ | $^oC$ | 104.68 | 113.73 |
| $T_{10}$ | $^oC$ | 111.15 | 115.81 |
| $\Delta T_9/dt$ | $^oC/s$ | 0.0252 | 0.0045 |
| $M_{init,9}$ | $mol$ | 8.65 | 9.06 |
| $M_{simp,9}$ | $mol$ | 1.5 | 1.5 |
| $M_{R,9}$ | $mol$ | 3.00 | 2.85 |

From simulation we get app. 200 ml (rectifier) and 300 ml (stripper) liquid on the trays, this give a total liquid volume in the column of approximate 2.7 liter, which is a rather good result compared to the dumping experiment $V_{l,d}$ and the computation based on the pressure drop.

## 5.2 Hydraulic time constant

### 5.2.1 Liquid

The hydraulic lag between the increase in reflux until the reboiler level changes is shown in Fig. 2 and found to be $\theta_M = 21s$. The response of the reboiler level to a change in the heat supply is shown in Fig.: 3. The operational parameters are stated in table 2.

Table 2: Experimental conditions of experiments 5 and 6 and the key parameters found

| | dimen. | Exp. 5 | Exp. 6 |
|---|---|---|---|
| $F$ | $ml/min$ | 350 | 350 |
| $z_F$ | | 0.56 | 0.45 |
| $L_{t=0}$ | $ml/min$ | 470 | 470 |
| $L_{t\geq 0}$ | $ml/min$ | 557 | 470 |
| $Q_{t=0}$ | $kJ/s$ | 5.8 | 6.5 |
| $Q_{t\geq 0}$ | $kJ/s$ | 5.8 | 8.0 |
| $\Theta_M$ | $s$ | 21 | 5 |

Applying Eq. 54 to the measured delay from changing the control signal of the reflux pump until the reboiler level start to change is incorrect. Inspecting Fig.: 2, it is seen that the experimental response is approximately 5 seconds delayed to the simulated response. We have to consider that it take app. 5 sec from changing the signal of the reflux pump until the flow changes. Take this into consideration, the liquid lag for the entire column is in the range of 16 seconds, which give an average hydraulic time constant of approximately $tau_l = 1.5s$. Applying Eq.: 38 to this set of operational conditions a time constant of 0.9 for the rectifier and 1.18 for the stripper is computed (see Tab.: 3)

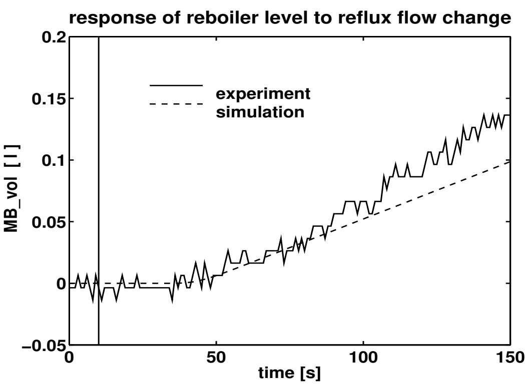


Figure 2: Response of reboiler level to a step in reflux flow at t = 20 s

The slightly different gradient in the response is due to the modelling of the bottom product valve. In the experiment the valve position is set, while in the simulation the volumetric liquid flow was set constant.

### 5.2.2 Vapor

Results of the estimation of $\tau_v$ from Eq. 37, $\tau_l$ from Eq. 38 and $\lambda$ from Eq. 45 for experiment 6 are shown in table 3. Parameters are computed for the initial flows stated in table 2. In Fig.: 4 the response of the tray temperature a step change in heat input is shown. The numerical results indicate an an inverse response for the simulation.

Table 3: Parameters estimated from data for Exp. 6 for $Q_{b,t=0} = 6.5 kJ/s$

| | | $Q_b$ | |
|---|---|---|---|
| | | rectifier | stripper |
| $M_{dry}$ | mol | 0.13 | 0.26 |
| $h_{dry}$ | mm | 34.4 | 34.4 |
| $M_{cl}$ | mol | 1.09 | 2.10 |
| $h_{cl}$ | mm | 9.8 | 18.7 |
| $\phi$ | | 0.23 | 0.49 |
| $\tau_v$ | s | 5.19 | 6.56 |
| $\tau_l$ | s | 1.18 | 0.99 |
| $\lambda$ | | -4.41 | -6.62 |

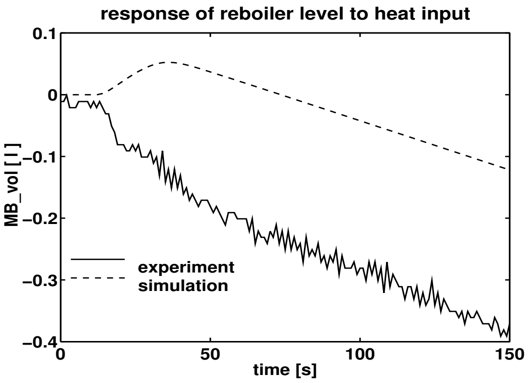


Figure 3: Response of reboiler level to a step in heat supply at t = 10 s

The simulated inverse response of the reboiler volume of maximum 0.05 liter corresponds to a change in the real reboiler of approximately $5mm$, which is approximately the resolution of the level measurement. The predicted change in the column holdup is app. 1.5 mol, which is less than 5 % of the reboiler holdup. Some liquid is pushed from the trays since the initial decrease in reboiler level slows down after 20 seconds. Nevertheless the extensive mismatch of experiment and rigorous model for the reboiler level has to be investigated and the model has to be corrected, it is not fully understood which effect of the real system is overestimated in the rigorous model.

## 5.3 Comparison of initial time constant of Experiment and Simulation

Step changes in the manipulated variables (reflux and heat input) were performed and the responses of the models and the real process are compared. The inventory of the distillation column is controlled by PI-controllers acting on the distillate and bottom flow, except for experiments 5 and 6 where the product flows are set manually. The presented responses are simulated with a tray efficency of 0.85 in the rectifier section and 0.8 for the stripping section. The efficiency of the reboiler is set to 0.6. The deviation from the steady state temperature is plotted to compare simulation by means of the rigorous model and the experimental data.

In **Experiment 2** the heat supply to the reboiler is increased at $t = 10s$. The temperature response of tray 3 and 9 is shown in Fig. 4.

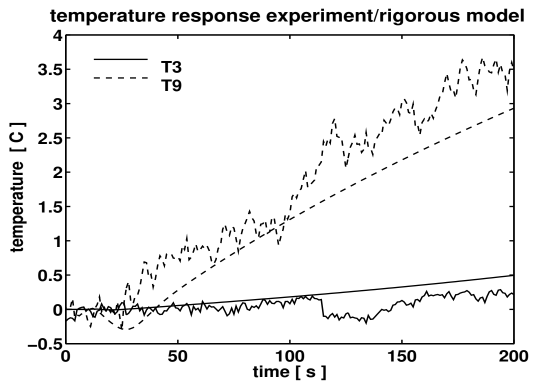


Figure 4: Initial response of temperatures on trays 3 and 9 to a step change in heat input at t = 10 s.

A thorough inspection of the first 100 seconds show an inverse response with a magnitude of $-0.25^oC$ in the response of the rigorous model. Since the noise level of the experimental data is $\pm 0.25^oC$ it is rather difficult to identify this from the set of experimental data. The experimental data where recorded at a sampling time of one second, without filtering of the signal, such that the inverse response should not be removed from the data set. Nevertheless the size and the duration of the inverse response is at a that high frequency that it is of only limited importance for control of the distillation column. Nevertheless it is not fully understood which effect of the real system was overestimated and initiate the inverse response.

The reflux flow is increased in **Experiment 3**, the temperature response of trays 3 and 9 are shown in Fig.: 5.

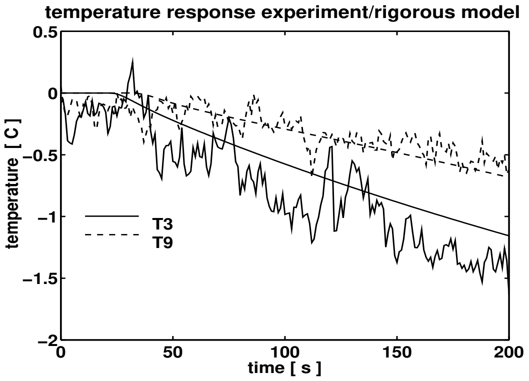


Figure 5: Initial response of temperatures on trays 3 and 9 to a step change in reflux flow at t = 20 s.

The responses of the rigorous model agree well with the recorded experimental data. The delays and initial time constants of the tray temperatures to the steps in heat input and reflux are in good agreement. In Fig.: 6 the response of the rigorous model (denoted with T3 R and T9 R) and the model with linearized tray hydraulics (T3 L, T9 L) are compared for a setp change of the reflux flow.

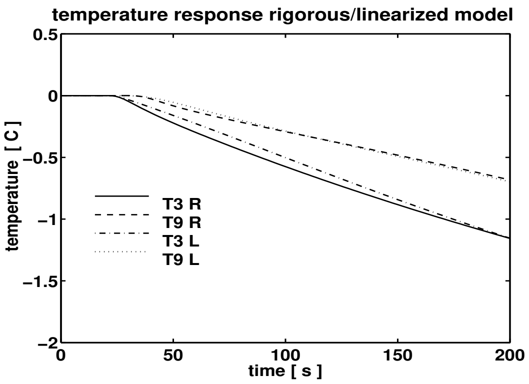


Figure 6: Initial response of temperatures on trays 3 and 9 to a step change in reflux flow. Step at t = 20 s.

The essential difference between the two models is the method used to determine the holdup and the tray hydraulics. Although the liquid holdup for the linearized model was determined from experiment an extensive mismatch in the initial time constant was observed (see Table 1). The hydraulic time constant $\tau_l$ and the vapor constant $\lambda$ are taken from table 3. Iteration on the liquid holdup in the linearized model to match the experimental initial time constants give a liquid holdup of 1.2 mol in the rectifier section and 1.5 in the stripper section. This holdup was used in to obtain the data shown in Fig.:6. The liquid holdup of the rigorous model is 2.8 and 2.85 mol in the rectifier and stripper respectively. The difference in holdup is approximately the amount of liquid in the downcomer.

# 6 Conclusion

The validity of the model has been demonstrated through the comparison of experimental and simulation results of the open-loop operation of the investigated distillation column. Nevertheless the model does not represent the reboiler holdup dynamics correctly.

It is fairly straight forward to generalize the results to other sieve tray distillation columns, since no for the investigated system specific correlations were utilized. The presented results demonstrate the importance of realistic modelled tray hydraulics for the proper operation of the process.

The transient responses of the hydraulic variables show the important role they have in the design process of a control system.

From the analysis of the simplified model we see that even if we use simple dynamic models for the composition dynamics the hydraulic dynamics should still be considered and modelled in an appropriate manner. Especially care has to be taken to obtain good estimates for the hydraulic time constants and the liquid holdup on the stage.

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## Notation

| | | |
|---|---|---|
| $A$ | active area | $m^2$ |
| $C_0$ | discharge coefficient | |
| $dp$ | dry pressure drop over tray | $N/m^2$ |
| $d$ | diameter | $mm$ |
| $F_i$ | molar feed flow of component $i$ | $kmol/h$ |
| $g$ | standard acceleration of gravity | $m/s^2$ |
| $h$ | molar enthalpy | $GJ/kmol$ |
| $h$ | height | $mm$ |
| $K_s$ | dimensionless velocity | |
| $L_i$ | molar liquid flow of component $i$ | $kmol/h$ |
| $M$ | molar holdup of liquid | $kmol$ |
| $MW$ | molecular weight | $kg/kmol$ |
| $p$ | pressure | bar, $N/m^2$ |
| $q$ | liquid flow | $m^3/s$ |
| $T$ | temperature | $^oC$ |
| $v$ | velocity | $m/s$ |
| $V$ | molar vapor flow of component $i$ | kmol/h |
| $y_{eq,i}$ | equilibrium molar fraction vapor | |
| $y_i$ | molar fractionof component $i$ | |
| $x_i$ | molar fraction of component $i$ | |
| $z_i$ | molar fraction of component $i$ | |

Greek Symbols

| | | |
|---|---|---|
| $\eta$ | Murphree tray effiency | |
| $\rho_m$ | liquid mass density | $kg/m^3$ |
| $\rho$ | liquid molar density | $kmol/m^3$ |
| $\phi$ | froth density | |

Subsripts

| | |
|---|---|
| active | active tray area |
| apron | downcomer apron |
| d,dc | downcomer |
| i | identifier |
| in | flow into the system volume |
| n | dummy index for number of components |
| l | liquid phase |
| out | flow out of the system volume |
| t | tray |
| v | vapor phase |