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DISTILLATION STARTUP OF FULLY THERMALLY COUPLED DISTILLATION COLUMNS: THEORETICAL EXAMINATIONS

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The fully thermally coupled distillation column offers an alternative to conventional distillation towers, with the possibility of savings in both energy and capital costs. This innovative and promising alternative provides the opportunity to separate a multicomponent mixture into fractions with high purities merely in one column. A lack of knowledge still exists when dealing with the startup of this process.

The startup of continuous distillation columns is a challenging control problem due to permanent changes in the process variables combined with highly nonlinear behaviour due to complex heat and mass transfer operation. The rigorous model presented in this contribution allows for a detailed analysis implying the startup behaviour of a distillation column from ambient and empty state to the steady state operating point. The model considers the influence of the heat accumulation of the column wall and internals and allows for a detailed analysis of variables with hydraulic and thermodynamic significance.

Beside this first time startup analysis of dividing-wall columns the designed model allows the solution of a wide variety of problems, from operability studies to process control.

KEYWORDS: distillation startup, dividing-wall column, rigorous process model, dynamic simulation

INTRODUCTION

Distillation remains the most commonly used separation process in the chemical industry, although one drawback is its significant energy requirement. When separating a homogenous multicomponent mixture into more than two products an arrangement with multiple distillation columns in series is traditionally realized. However, especially the separation of a multicomponent mixture in a sequence of distillation columns is a high energy consumer. In that respect, the use of complex distillation arrangements, such as the fully thermally coupled (Petlyuk) configuration, leads to significant reduction in energy consumption compared to conventional arrangements. The Petlyuk Column is thermodynamically preferable as backmixing of the intermediate product is avoided due to prefractionation of the feed. Theoretical studies (e.g. Petlyuk et al. 1965, Kaibel 1988, Fidkowski and Krolikowski 1989) have revealed that this column configuration is capable of achieving 30% of energy savings.

Dedicated to Prof. Dr.-Ing. W. Roetzel on the occasion of the 70th anniversary of his birthday.

Finally, proceeding on the path of process integration leads to the dividing-wall column (Wright 1949). This concept can be achieved by arranging a vertical partition in the middle of a column (Kaibel 1987). Therefore, the difference between a conventional distillation column and a dividing-wall column refers to the column internals. As the vertical partition separates the column into a feed and a side stream section, dividing-wall columns are thermodynamically equivalent to Petlyuk columns. The dividing-wall avoids radial mixing of vapour and liquid streams and enables the withdrawal of three products with any purity in one column. A dividing-wall column is especially favourable in case a high-purity middle product is required. Nevertheless, even in case a simple side stream column for removing the intermediate product is applicable, a dividing-wall column might be advantageous due to lower column height and less energy requirements.

Although theoretical studies have demonstrated the economic advantages of dividing-wall columns for the mentioned scenarios, industry hesitated to build the columns. The lack of knowledge about design and control of these columns has prevented their application in industry for a long time. In recent years several academic groups have researched this area (Wolff and Skogestad 1995, Serra et al. 1999, Halvorsen and Skogestad 1999). One group set up a pilot-scale column to study controllability and operability (Mutalib and Smith 1998a, Mutalib et al. 1998b). This work has contributed to a better understanding of the design and control and therefore to an increasing acceptance of dividing-wall columns within industry. Recently an advanced process control concept has been successfully applied to a pilot plant column (Adrian et al. 2004). In connection with a growing demand for high-purity products in fine and commodity chemistry within the last years, dividing-wall columns arise more and more interest.

A special problem, which has not been considered yet, is the startup process of dividing-wall columns. Systematic, practically feasible and time optimal startup control approaches are definitely desired, which guarantee safe plant operation. Hence, theoretical examinations aiming at time optimal startup strategies provide an excellent performance, as they can be conducted fast, cost-efficient and sufficiently detailed. Nevertheless, the startup of continuous distillation columns is a challenging control problem in the industrial practice. This implies permanent changes in the process variables combined with highly nonlinear behaviour due to complex heat and mass transfer operation. Some authors dealt with detailed analysis of the startup phase for conventional distillation columns (Ruiz et al. 1988, Elgue et al. 2004, Wozny and Li 2004). A detailed analysis for the startup of fully thermally coupled distillation columns has not been examined so far. All different startup models are based on a rigorous tray-by-tray approach, whereas the startup behaviour is characterized as a discrete process from non-equilibrium to vapour liquid equilibrium state.

Elgue et al. 2004 pointed out, that the temperature profile is characteristic for the development of hydrodynamic and thermodynamic variables during startup phase. Furthermore, Figure 1 sketches the discontinuities, which occur when analysing the startup of a distillation column from ambient and empty state.

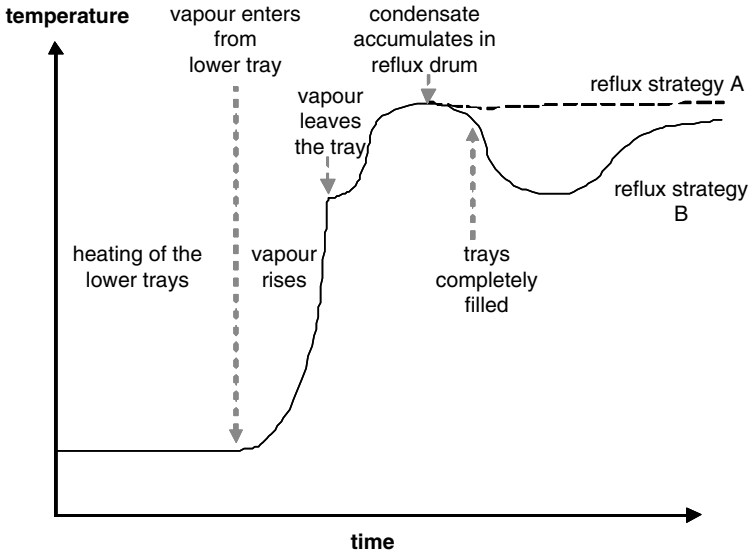

Figure 1. Temperature profile during startup operation in a distillation column

RIGOROUS PROCESS MODEL

The theoretical examinations in this contribution are based on a rigorous process model, which describes the physical and thermodynamic phenomena in a fully thermally coupled distillation column. As a packed distillation column is simulated, the HETP value provided by the manufacturer is used to discretise the internal packings into single equilibrium stages (Figure 2).

The following assumptions are made in the development of the model: the molar vapour holdup is negligible since vapour density is considerably smaller than liquid density; perfect mixing on each stage is assumed in order to reduce complexity of the problem; a tray efficiency factor can be introduced on each stage in order to consider differences from phase equilibrium.

The model consists of ordinary differential equations obtained from mass and energy balances around each stage and a set of algebraic equations which are used to predict the physical properties, the vapour-liquid equilibrium and the column hydrodynamics. The index i denotes the theoretical stages numbered from top to bottom, whereas index j refers to the components.

Component mass balance:

$$\frac{d(HU_i x_i^j)}{dt} = L_{i-1}x_{i-1}^j + V_{i+1}y_{i+1}^j - L_i x_i^j - V_i y_i^j \qquad (1)$$

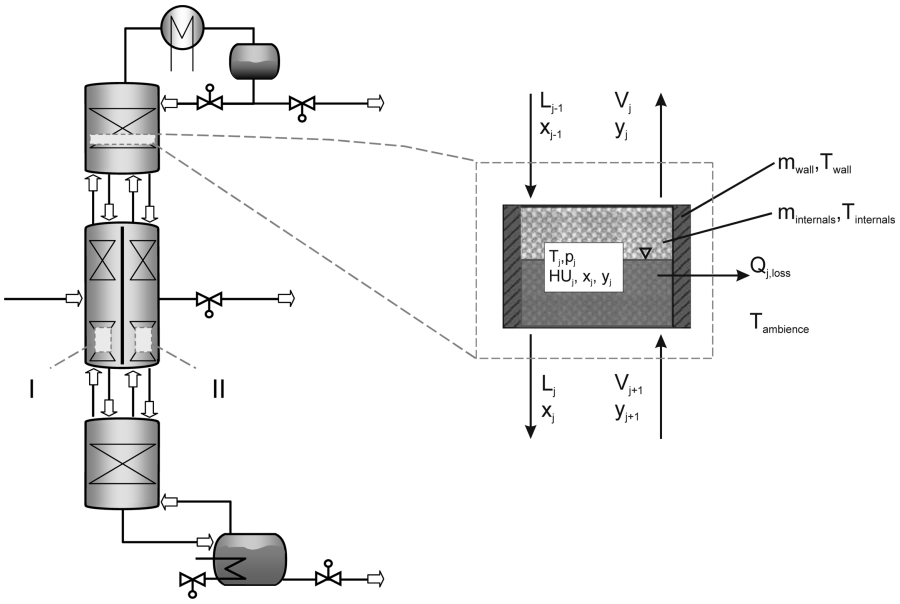

Figure 2. Scheme of the process model and the general stage model

Overall mass balance

$$\frac{dHU_i}{dt} = L_{i-1} + V_{i+1} - L_i - V_i \tag{2}$$

Energy balance

$$\frac{d(HU_i h_i^{liq})}{dt} + \frac{d(mc_p T)_{i,internals}}{dt} + \frac{d(mc_p T_m)_{i,wall}}{dt} = L_{i-1}h_{i-1}^{liq} + V_{i+1}h_{i-1}^{vap} - L_i h_i^{liq} - V_i h_i^{vap} - Q_{i,loss} \tag{3}$$

with

$$T_{i,m} = \frac{T_i + T_{ambient}}{2} \tag{4}$$

Vapour-liquid equilibrium

$$y_i^j = K_i x_i^j \tag{5}$$

Summation equation

$$\sum_j y_i^j = 1 \tag{6}$$

Heat loss

$$Q_{i,loss} = kA(T_i - T_{ambient}) \tag{7}$$

Pressure model

$$p_i = p_{i-1} + \Delta p_i \text{ with } \Delta p_i = f(\text{geometry, physical properties, tray hydraulics}) \tag{8}$$

Holdup model

$$HU_{i,max} = f(\text{geometry, physical properties, tray hydraulics}) \tag{9}$$

Pressure drop Δp_i and maximum holdup HU_{max} can be calculated from various correlations published by Engel et al. 2001 and Mackowiak 2003. Simulation studies have shown, that previous calculation of the maximum holdup improves the robustness during simulation without losing preciseness.

This highly nonlinear differential algebraic equation system is implemented in the commercial software tool Aspen Custom Modeler™. The model gets the required physical and thermodynamic properties of the components via interface from Aspen Properties™. Beside equilibrium stages, the entire column model contains a total condenser with adjustable subcooling, a reflux drum and a reboiler (Figure 2).

So far, the model is applicable for operability studies in closed-loop mode. The software tool Aspen Custom Modeler™ offers control elements for operation control of dynamic processes, respectively. When dealing with the startup behaviour of distillation columns from ambient conditions the rigorous process model has to consider the following aspects additionally: variable vapour flow, heat accumulation of the column wall and the column internals, variable liquid flow and non-adiabatic operation.

In case of examining high purity distillation columns the model inherently contains a differential equation system of high stiffness due to the different dynamics of overall and component mass balances. Considerations of the startup behaviour from ambient conditions imply the occurrence of discontinuities in the process variables if vapour or liquid enters an empty stage. These discrete-continues transitions in the process combined with the high stiffness of the system demand for efficient and robust numerical solver which are the basis for obtaining successful and reliable solutions. Application of an implicit solver, e.g. Gear-Algorithm or implicit Euler Method, avoids numerical problems. Gear's algorithm and Newton's method provide sufficient accuracy and have been chosen to solve the DAE system. Explicit numerical solvers are not appropriate as to little step size is required in order to guarantee stability. An elaborated model structure is furthermore required to avoid index problems.

SIMULATION RESULTS

The theoretical examinations in this contribution are part of an integral analysis of the transient behaviour of dividing-wall columns. As a stainless steel dividing-wall distillation column with a diameter of 68 mm is currently set up in our laboratory, the process model considers the peripheral devices and geometric parameters of this pilot plant: a total condenser, a reboiler, a reflux drum and column internals consisting of 980 mm Montz BSH-500 packings in each case. At first, the conclusiveness of the process model with regard to the physical phenomena in the column is verified.

As dividing-wall columns are operated continuously, it is important to distinguish between two different tray hydraulic scenarios. In that respect, Figure 3 visualises that some trays are filled because of the downcoming liquid feed stream (Phases A-B), whereas others exceed HU_{max} due to condensation effects of upcoming vapour (Phases 1–2). The scenario depicted on the left corresponds to the physical phenomena in the lower feed section (packing section I in Figure 1). Accordingly, the hydraulic processes in the lower side stream section (packing section II in Figure 1) are considered on the right.

Initially, the column is empty and at ambient conditions. The preheated liquid feed L_{i-1} enters the column and the i^{th} tray is filled up to its maximum holdup HU_{max} (Phase A). In case HU_{max} is exceeded, a liquid steam L_i leaves the tray (Phase B) and runs down the column. Phase A and B repeat until the liquid arrives at the reboiler. A minimum liquid level in the reboiler is required before the heating is activated. The rising vapour stream

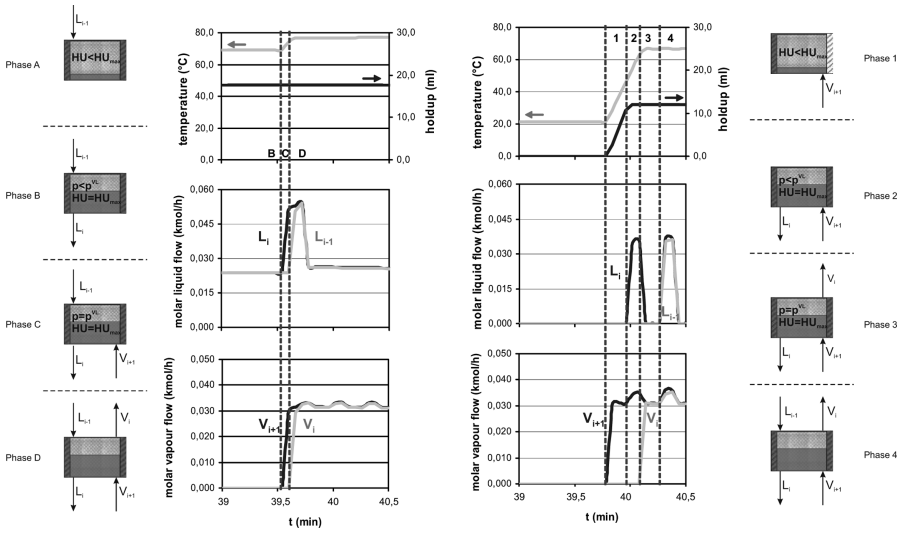

Figure 3. Hydraulics of trays in the lower feed section (left) and of trays in the lower side stream section (right)

V_{i+1} condenses and the liquid outlet flow L_i increases immediately, which is indicated by Phase C. The rising vapour heats the tray until the liquid exceeds boiling temperature. Then, the vapour-liquid equilibrium is computed. If p_{vp} is greater compared to the pressure p on the tray, a vapour stream V_i leaves the tray (Phase D).

Consequently, the rising vapour streams enter stages, which are still empty. In this case vapour stream V_{i+1} is condensed at the column wall and the internals (Phase 1). Hence, the tray is filled until its HU_{max} is exceeded. After the liquid holdup HU has exceeded HU_{max}, a liquid stream L_i leaves the tray (Phase 2). The upcoming vapour stream V_{i+1} continues heating the tray until the liquid reaches boiling temperature and a vapour-liquid equilibrium is calculated. Phase 3 starts, if the vapour pressure p_{VL} exceeds the pressure p on the tray. Thus a vapour outlet stream V_i is calculated. In case liquid from the upper tray flows downwards, Phase 4 has begun. The heating process in the feed section occurs faster than in the side stream section because the trays are already filled and preheated by the feed stream.

The simulation results demonstrate the conclusiveness of the developed model as the previously described phenomena can be explicitly identified.

The next step is the simulation of the entire startup behaviour of a dividing-wall column in order to emphasise the physical capability of the process model. The temperature profiles are typically characteristic for discrete-continues changes of the hydrodynamic and thermodynamic process variables during the startup phase. Therefore, the separation of an industrially relevant ternary mixture of n-hexanol, n-octanol and n-decanol into high purity products (>99 weight-%) has been simulated. The process is run under vacuum conditions at a condenser pressure of 15 mbar. Figure 4 depicts the

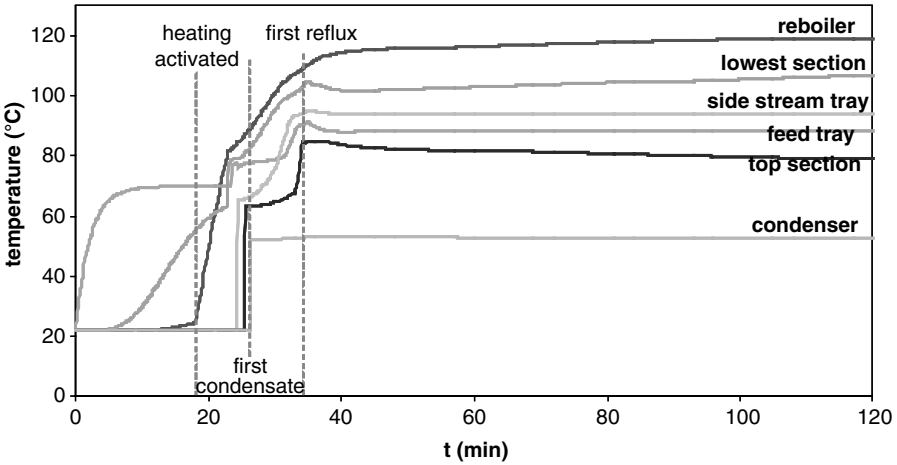

Figure 4. Temperature profiles during startup of a dividing-wall column

temperature profiles for the startup process of a dividing-wall column. Exemplarily, trays in each column section are chosen. The different phases can be expectedly identified.

In this simulation run all manipulated variables are set to their steady state values. This startup strategy is widespread in the industrial practice. Nevertheless, it takes about 360 min until the column is run at the desired steady state operating point, which guarantees the required high purity products. This definitely demands for developing appropriate startup strategies in order to shorten unproductive startup time by far.

CONCLUSION

This contribution deals with first time startup analysis of a fully thermally coupled distillation column. The conducted theoretical examinations consider the startup behaviour of a column from empty state and ambient conditions. This requires an elaborate process model and robust numerical solvers. The simulation results contribute to an advanced understanding of the physical phenomena in dividing-wall columns. The model has been validated with respect to its physical conclusiveness. An equivalent pilot plant will be set up soon in order to validate the process models with experimental data. The rigorous process model serves as a promising basis for developing and applying time optimal startup strategies.

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