COMBINING CONCEPTUAL AND DETAILED METHODS FOR BATCH DISTILLATION PROCESS DESIGN

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Abstract

The separation of multicomponent mixtures by batch distillation generally requires the sequential processing of several splits in one column. The sequencing of the splits and the choice of the column configuration, e.g. regular or inverse column, has a substantial impact on the economic potential of the process. Beside these structural design decisions the profitability of the process is further governed by the transient profiles of the control variables, mainly the vapor boil-up rate and the reflux ratio. The large variety of structurally different operational strategies results in a computationally demanding discrete-continuous dynamic optimization problem. In this contribution, we present how a fast conceptual design method based on the rectification body method (RBM) for the design of continuously operated columns can be utilized to significantly improve the performance and robustness of rigorous optimization-based design methods. It is used to exclude economically unattractive splits from the superstructure for the dynamic optimization problem and to provide an initialization for the dynamic optimization including time-varying initial profiles for the control variable profiles. The approach is illustrated with a zeotropic quaternary mixture.

Keywords

Batch distillation design, Geometric design method, Rectification body method, RBM, Discrete-continuous dynamic optimization, Superstructure model, Initialization.

Introduction

Multicomponent batch distillation is frequently encountered in the specialty chemicals and pharmaceutical industries. Classical operating policies are characterized by either a constant reflux strategy with variable product composition or a variable reflux strategy where the key component in the product is held at constant composition. The most profitable batch operation is obtained when the distillation column is operated under optimal reflux. Besides the flexibility in terms of operational strategies, batch distillation processes allow for a number of different column configurations, such as regular, inverse, middle or multi-vessel designs (Kim and Diwekar, 2001). Multicomponent mixtures can be processed in one single batch distillation column that is operated in multiple process stages. The sequencing, i.e. the chronological order of splits processed in a batch distillation column, is not necessarily fixed a-priori and can thus be used as structural design degree of freedom to improve the process performance. If, in addition, several column configurations are considered for each stage of the batch distillation process, we end up in a design problem with a combinatorial character (Oldenburg et al., 2003). Together with the time-dependent continuous decision variables on each stage, the optimal design problem results in a discrete-continuous dynamic optimization problem that can be very challenging to solve.

Alternatively, conceptual design methods can be used to find the most promising sequencing of a batch distillation process including the configuration of each process stage. In this work, a novel method is proposed for the conceptual design of multicomponent batch distillation processes that provides a deep insight into the separation task under consideration. This method can either be used as a stand-alone design tool, or as part of combined conceptual and detailed design method. In this contribution, we focus on a combination of this conceptual design method with a detailed design method that is based on the discrete-continuous dynamic optimization of rigorous superstructure models. It will be shown how the dynamic superstructure can be simplified using the results of the conceptual design method. Moreover, the initialization of the dynamic optimization subproblems can be significantly improved.

The overall procedure will be illustrated by the separation of a zeotropic quaternary mixture of 100 kg n-pentane, 100 kg n-hexane, 600 kg n-heptane and 200 kg n-octane into the respective pure components with a purity of 99%. For each batch configuration the optimal trajectory of the product flowrate minimizing the time of operation is to be determined for a fixed reboiler heat duty Q_B=50 kW.

Conceptual Design Procedure

Shortcut design methods for the determination of the minimum reflux ratio of continuously operated distillation columns have been a workhorse for process synthesis since the first introduction of the method of Underwood (1948) for ideal mixtures. In the last years, several authors have introduced so-called geometric design methods (see Bausa et al., 1998, for a review) that extend the determination of the minimum energy demand to nonideal mixtures.

In principle, these methods can be successfully adapted to the design of batch distillation processes if the holdup in the still is significantly larger than the holdup on the trays of the column. In good approximation the profiles can then be regarded as quasi-stationary. First publications on the application of geometric design methods for batch distillation for ideal mixtures are reported by Salomone et al. (1997) and Lotter and Diwekar (1997). Espinosa and Salomone (1998) use pinch theory in order to generalize the geometric design to nonideal ternary mixtures. Their method however requires a-priori knowledge of the active pinch points for which the method is to be applied.

In contrast to a steady-state column the reflux ratio, the composition in the still and therefore the column profiles change with time resulting in a transient trajectory of different steady states. The change of the still composition $\mathbf{x}_N$ and holdup N with time result from the material balance around the column

$$\frac{dN}{dt} = -S \qquad (1)$$

$$\frac{dx_{N,i}}{dt} = -\frac{S}{N}\left(x_{S,i} - x_{N,i}\right) \qquad i = 1,\ldots,C \qquad (2)$$

where N denotes the still and S the product stream. Figure 1 illustrates the corresponding balance envelope if the column is operated as a regular column. The transient profiles for still holdup and composition can easily be obtained by integration if S and $\mathbf{x}_S$ are known at each time step. The strategy for obtaining this necessary additional piece of information depends on the desired type of split. The two most important types are the direct and indirect split. Note that in the following a regularly operated column will always be assumed. The application of the method for an inverse column is done analogously.

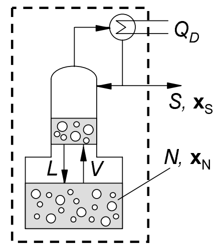

Figure 1. Balance envelope for a regular column

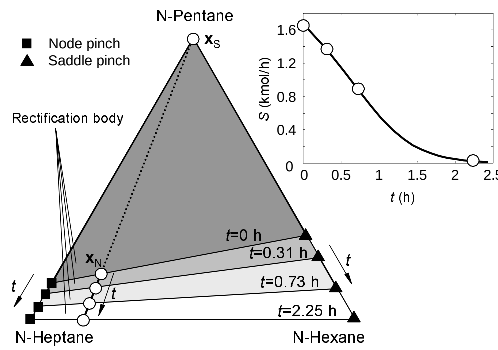

Figure 2. Rectification bodies for direct split

Direct Split

The direct split is characterized by the recovery of a conceptually time-invariant product composition $\mathbf{x}_S$. Often $\mathbf{x}_S$ will correspond to a pure component as for the overhead separation of pentane from hexane and heptane that will serve as an illustrative example in this section. Since we assume quasi-stationary operation the column profile can be described using the concept of tray-by-tray calculation. Feasible operation of the column is then subject to finding a profile that connects $\mathbf{x}_S$ and $\mathbf{x}_N$. In order to avoid solving the rather difficult problem of intersecting a line and a point in two-dimensional space, a feasibility check based on pinch theory is used. Here, we resort to the rectification body method (RBM) of Bausa et al. (1998) which approximates the manifold of potential profiles by geometrical rectification bodies based on the pinch points. It is applicable to arbitrary multicomponent mixtures. The RBM procedure links the feasibility of obtaining the prespecified $\mathbf{x}_S$ to the reboiler heat duty Q_B and the rate S of product withdrawal. For a given Q_B the maximum $S=f(\mathbf{x}_S,Q_B)$ can therefore be found. Figure 2 illustrates the formation of the rectification bodies at four discrete time steps. The corresponding trajectory of S is also shown. Note that the decision to use RBM was made solely because of its generality. For the simple ideal mixture discussed above any other geometric design method would be identically suitable.

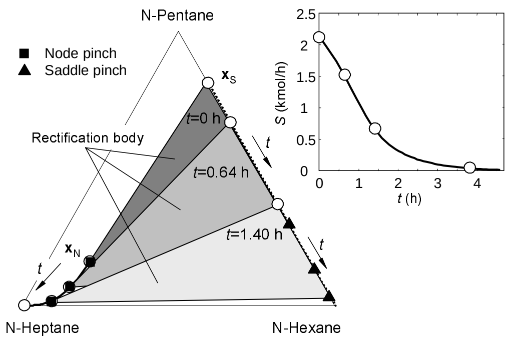

Figure 3. Rectification bodies for indirect split

Indirect Split

In the indirect split the desired cut is the final contents of the still $\mathbf{x}_N(t_f)$. An example is the recovery of heptane from the ternary mixture already discussed above in a regular column. During operation a time-variant product composition $\mathbf{x}_S(t)$ will be withdrawn. Conceptually, $\mathbf{x}_S$ will be located on the edge of the distillation region in which the process is performed. Since the example mixture is zeotropic the edges of the distillation region coincide with the edges of the composition space. Therefore $\mathbf{x}_S$ will be essentially free of heptane at any time step

$$0 = x_{S,k} \qquad k = \text{Heptane} \quad (3)$$

As for the direct split, $\mathbf{x}_S$ and $\mathbf{x}_N$ must be connected by the column profile. The maximum product flowrate is obtained if $\mathbf{x}_N$ is a stable node pinch with respect to $\mathbf{x}_S$ complying with the pinch equations

$$0 = L - V + S \quad (4)$$

$$0 = Lx_{N,i} - Vy_{N,i} + Sx_{S,i} \qquad i = 1,\ldots,C \quad (5)$$

$$0 = y_{N,i} - K_{N,i}\left(\mathbf{x}_N, T_N\right)x_{N,i} \qquad i = 1,\ldots,C \quad (6)$$

$$0 = Lh_N^L\left(\mathbf{x}_N, T_N\right) - Vh_N^V\left(\mathbf{y}_N, T_N\right) + Q_B \quad (7)$$

(Bausa et al., 1998). Eqs. 4-7 have 2C+3 equations in 2C+4 unknown variables ($\mathbf{x}_N$, $\mathbf{y}_N$, T_N, L, V, S). The location of $\mathbf{x}_S$ as given by Eq. 3 fixes the remaining degree of freedom. Figure 3 shows the rectification bodies and the profiles of $\mathbf{x}_S$ and $\mathbf{x}_N$ over time in a ternary diagram.

Note that this design method for the indirect split can in principle also be applied to arbitrary azeotropic mixtures. In this case however, the edge of the distillation region may not fall together with an edge of the composition space and a suitable function $f(\mathbf{x}_S)=0$ for characterizing the distillation boundary must be known. Furthermore, the method must be extended by a suitable algorithm for the handling of tangent pinches which may appear for strongly nonideal mixtures.

Detailed Design Procedure

Methods for detailed batch distillation design rely on a mathematical optimization of the process design and operation subject to dynamic column models which are usually based on the MESH equations (Kim and Diwekar, 2001). Such an optimization-based design method is very flexible since it does not depend on any simplifying assumption and a variety of different design goals can be achieved by specifying an appropriate objective functional and constraints. For multistage batch distillation design, as considered in this paper, the optimization problem results in a discrete-continuous dynamic optimization problem due to the type of degrees of freedom occurring. Besides the continuous time-variant decision variables S as well as the operating time required to produce one split, discrete decision variables are present since the mode of operation (i.e. regular or inverse) in each process stage and the sequencing of the splits are not fixed a-priori. Solving a discrete-continuous dynamic optimization problem is a challenging task due to its combinatorial complexity in conjunction with the nonconvexity of the dynamic model and the constraints. The determination of a 'good' suboptimal solution with a local optimization technique is difficult since (i) the search in the discrete decision variables is terminated on the basis of heuristic stopping criteria, possibly at an arbitrary local solution, and (ii) the dynamic optimization subproblems are difficult and time-consuming to solve.

Combination of Conceptual and Detailed Design

Having outlined the basic properties of the conceptual and the detailed design method, we now present how these two different methods can be combined in order to more efficiently and reliably determine an optimal or at least a good suboptimal separation process design and operation. The potential of this combination will be highlighted using the quaternary example process for which an optimal design and operation was presented by Oldenburg et al. (2003) on the basis of a full dynamic superstructure model.

Particularly, the conceptual design method is used for rapid screening of all possible product splits in conjunction with either a regular or an inverse operation. Depending on the separation task, i.e. the initial amounts as well as the desired product purities, such a screening can yield an approximate classification into splits that are favorable and unfavorable. Considering all possible product split options as well as the mode of operation in each stage of the separation, i.e. regular or inverse operation, we end up in 32 different configurations with differing batch times (see Figure 4 for a comparison of the batch times calculated with both methods). It is found that the removal of high purity octane in an inverse column leads to excessively long batch times. With this insight from the conceptual model, the respective 16 splits can be removed from the superstructure of economically relevant alternatives effectively cutting the necessary combinatorial effort of the detailed model in half. However, it can also be seen that the accuracy of the conceptual method is insufficient to discriminate between the other 16 alternatives which only show small differences in total batch time.

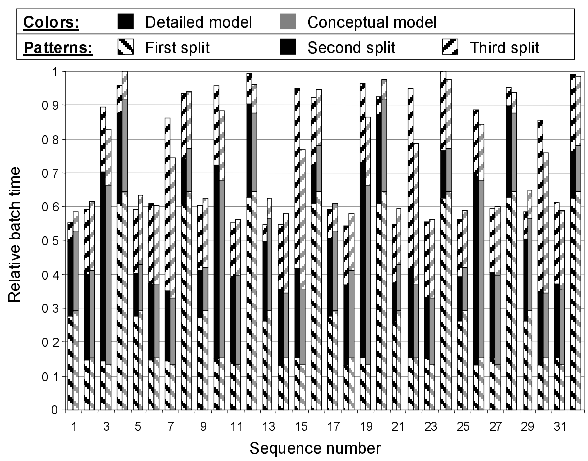

Figure 4. Comparison of batch times

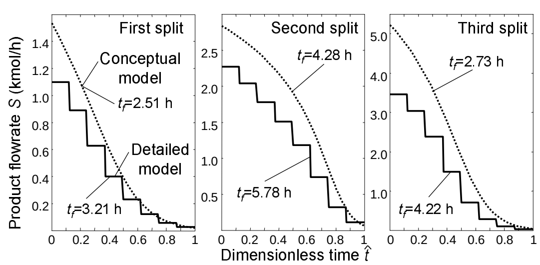

Figure 5. Batch times and control profiles

Even with a reduced integer search space, the successful application of any mixed-integer dynamic optimization technique largely depends on the effort spent for solving dynamic optimization subproblems with either fixed or relaxed discrete decision variables. For an excellent overview of the corresponding solution methods see Grossmann (2002) and Floudas (1995). In particular, it is not trivial to find an initial guess which at least guarantees feasibility. One strategy to find an appropriate starting point for the multistage problem is to decompose the multistage problem into a set of single stage problems for which convergence is obtained easily. It was found that this rather tedious procedure can be circumvented by directly exploiting the existing data available from the conceptual design method. Using the respective control profiles and the batch times convergence was obtained without any decomposition strategy. Figure 5 shows the good agreement of the control profiles of the conceptual with the time-optimal control profiles calculated with the detailed design approach. It can be seen that the conceptual model predicts slightly higher product flowrates. This can be partly attributed to the underlying assumption of a column with an infinite number of trays whereas the detailed model considers a column with 10 trays.

Conclusions

The design of a quaternary zeotropic batch distillation process using a conceptual and a detailed design model has been presented. It has been shown that while both methods can be applied independently their true power only unfolds when they are used in combination. The accuracy of the conceptual design method is insufficient for correctly finding the economically most attractive alternative. It can be used, however, to significantly reduce the size of the combinatorial problem by removing undesirable splits from the superstructure and to provide a good initial guess. Using this initial guess, rapid convergence of the detailed model to the optimum is achieved. The combination of conceptual and detailed models can therefore be a means to reduce convergence problems and computation time which currently prevents large-scale application of detailed design methods in industry.

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