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## Minimising the startup duration for mass- and heat-integrated two-column distillation systems: A conceptual approach

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#### Abstract

The startup of a chemical plant is a complex and highly dynamic process. The main source of complexity stems from the simultaneous variation of all process variables until the required operating point is reached. In previous studies, the startup of single columns for conventional and reactive distillation was considered. A two-column system was also analysed, but featuring only heat coupling without mass integration between the columns, which means with no positive feedbacks. In this work, a heat- and mass-integrated two-column system for continuous pressureswing distillation (PSD) is investigated experimentally and theoretically on the example of the acetonitrile-water mixture. Rigorous models of the individual equipment units such as trays, condensers and heat exchangers have been formulated and integrated in an overall dynamic system model which has been validated against experimental data. Further, a simple qualitative model to guide the minimisation of the startup duration has been derived and applied to the considered distillation system. The results show that, following the derived criteria, it is possible to reduce the startup duration for the example in Case Study 1 by 78% compared to the duration of empirical startup procedure. © 2007 Elsevier B.V. All rights reserved.

Keywords: Process startup; Startup optimisation; Pressure-swing distillation; Heat- and mass-integration; Homogeneous azeotropes

#### 1. Introduction

The separation of homogeneous azeotropic mixtures is a very common process in chemical industry, frequently employed for solvent recovery. To solve the separation problem, pressureswing distillation [1], can be an efficient alternative to the broadly applied azeotropic and extractive distillation processes. Only few theoretical results on this process have been published and experimental data are also scarce [2], which seem to be the reasons for its rare industrial application. An exception to this is the separation of tetrahydrofuran (THF)–water mixtures, for which pressure-swing distillation is the method often used by industry [1]. The main advantages of PSD are the improved energy efficiency and the elimination of entrainers. On the other

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hand, the material and energy integration of the columns result in a highly complex plant which is more difficult to control [3,4].

From the perspective of system operation, one of the critical phases is the system startup. This is an inherently non-productive phase involving only costs and bringing no direct benefits. Columns in industry are often used for different products. Therefore, shutdown and startup operations are regularly performed. Although startup is not the most time-consuming period in the operation cycles, its duration is not negligible. The startup of a coupled column system like the process considered in this contribution is a very complex and highly dynamic procedure. A decrease by 50% of the startup duration can be in the range of days for a big industrial system and this can save significant resources-most notably energy, as well as reduce waste and ultimately, costs.

Startup is usually performed only with no or little modelbased optimisation. The prevailing practices are based on operator experience rather than conceptual understanding. As a result, startups tend to be time-consuming, leaving a large scope for reduction of their duration and costs. This problem is especially complex for systems integrating more than one distillation

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column, which is the case with pressure-swing distillation. The increased complexity in this case stems from the need to carefully coordinate the flow-rates of the feed, reflux and recycle streams as well as the rate of utility heating.

A simulation model of a two-column PSD system considering established operation in continuous mode has been proposed by Repke et al. [5]. This model was used to analyse the robustness of the system operation towards disturbances. Repke et al. [6] present a model for startup simulation of a batch distillation column on the example of the acetonitrile—water mixture. In that work two groups of equations are defined and are switched when the tray reaches bubble point. Wendt et al. [7] report an optimisation study of the startup of a heat-integrated two-column system for water—methanol separation. In this case, the two-column arrangement is used merely to achieve energy savings by means of heat-integration and no mass integration is applied.

One of the works introducing conceptual insight into the behaviour of distillation columns during startup is that of Ruiz et al. [8], which uses the generalised distillation columns model developed in the first part of the same series of papers [9]. This work identified three major phases during column startup: discontinuous, where all variables change their values drastically; semi-continuous, during which the hydraulic variables reach their steady-state values; and finally a continuous phase, during which the thermodynamic variables also reach their steady-state values. As the authors point out, the latter phase is also the lengthiest one.

Sørensen and Skogestad [10] investigated the startup procedures for batch distillation processes. Their main motivation was that in batch operations the startup times may well last as much as 50% of the total operating time. The authors considered two options for improving the procedures for batch distillation startup—partial backmixing and reusing light component in the condenser drum from a previous batch.

A more recent work by Fabro et al. [11] proposes using an advanced combination of control thechniques, including neural networks, fuzzy controllers, both managed and adapted by using optimisation via a genetic algorithm.

The discussed works [8,10,11] use optimisation models for improving the startup of single distillation columns operating in batch or continuous mode. They, however, do not consider the effects of mass- and heat-integrating two distillation columns. Also, no physical insight is provided there of the dependencies between the values of the manipulated variables and the controlled ones. As it will become apparent, the speed of startup is inherently dependent on the combined values of the flow-rates of the streams external to the system and the reboiler duties.

In the current work, a detailed dynamic model of a twocolumn system for continuous PSD is first presented, which is based on the previous works by Repke et al. [6] and Wang et al. [12]. The system is considered started when the product streams reach the quality specifications. It is shown that by applying simple conceptual insight, revealing the limitations on the flow-rates of the feed and the distillate recycles, it is possible to reduce the startup times significantly, by almost one order of magnitude.

The particular component system used in the experiments and the calculations is acetonitrile—water. Acetonitrile—water is a

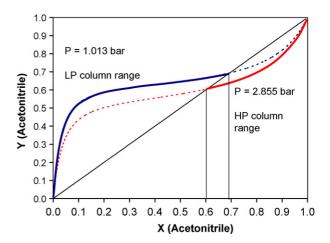


Fig. 1. Acetonitrile-water VLE diagram for two different pressures.

binary mixture, featuring a pressure-sensitive azeotrope. A VLE diagram for two example pressures is shown in Fig. 1, where the regions of operation of the low-pressure (LP) and high-pressure (HP) columns are also indicated. The system flowsheet is given in Fig. 2. The feed, depending on its composition, is introduced into the system as follows. Feed streams with lower acetonitrile content are introduced into the low-pressure (LP) column and acetonitrile-rich streams to the high-pressure (HP) column, respectively. In any of the cases, acetonitrile is obtained as the bottom product of the HP column and water as the bottom product of the LP column. The distillate of each column is recycled to the other one several trays above the corresponding feed tray. The mole fractions of the distillates are close to the azeotropic compositions at the respective pressures. The considered column system is also heat-integrated by condensing the HP column distillate against the evaporation of the mixture in the LP column reboiler (Fig. 2). Two case studies have been performed with the feed mixture containing 27 mol% (0.27 mole fraction) and 40 mol% (0.40 mole fraction) acetonitrile, respectively.

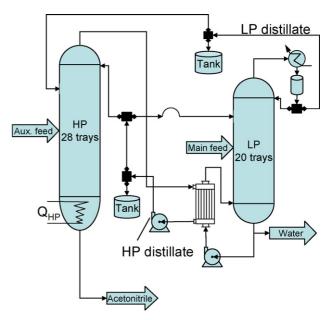


Fig. 2. Flowsheet of the analysed system.

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