# Abstract

Chromatography is a powerful separation technique mainly used for difficult separations of temperature-sensitive substances or substances with similar properties that are difficult to separate by other methods. Among the various chromatographic processes, the simulated moving bed (SMB) process, which is the continuous implementation of the basic chromatographic process, has found great practical application.

The SMB process allows for significantly higher productivity than the chromatographic batch process while simultaneously reducing solvent consumption, thereby lowering the cost per unit of production. Its main disadvantage is its complexity. The process is inherently nonlinear, both due to the chromatographic system involved, consisting of the solutes, the adsorbent and the desorbent, and the nature of the interaction between them. Furthermore, at their economic optimal operating point, SMB processes are highly sensitive to disturbances. In practice, SMB processes are most often operated in an open-loop mode, with the selected operating point not at the optimum but at a suboptimal one located in an operating region with higher robustness to disturbances but reduced productivity. That is why the use of automatic control in SMB operations is particularly attractive.

From a control perspective, SMB processes are very challenging – they are characterised by complex hybrid dynamics including cyclic operation, long settling times, interconnected control channels, and limited measurement information. That requires a deep understanding of the process. Mathematical modelling and computer simulations are indispensable tools for the design, optimisation, and control of such processes, which are also used in this work.

This thesis builds upon the research on the control of SMB processes first carried out by Marco Fütterer (Fütterer, 2010) and then further continued by Paul Suvarov (Suvarov, 2016) in Magdeburg and Mons. They proposed a simple yet powerful control strategy, which was successfully validated experimentally by Suvarov, for SMB processes operating with adsorption isotherms in the linear region of the isotherms. At the same time, this strategy showed some weaknesses when operating in the nonlinear region of the isotherms, which is the main focus of the current thesis, particularly for chromatographic systems with Langmuir isotherms.

The pilot plant considered has four controlled variables – the two product purities and two dimensionless retention times to control the total regeneration in the outer zones. The manipulated variables are the flowrates in each of the zones of the SMB plant. The original control concept of Suvarov is first further extended by adding two decouplers to reduce cross-coupling in the product control channels, thus providing a decentralised control scheme. Moreover, to achieve a faster response, the PI controllers are replaced with PID ones. The results showed that this control strategy performs very well around the operating point at which the controllers and decouplers are tuned. However, performance deteriorates when not operating close to it. Moreover, the capability of handling input constraints is limited.

Therefore, a different control strategy based on online optimising control was developed. In particular, a novel semi-centralised control scheme is proposed in which the two product purities are controlled by a nonlinear model predictive controller, whereas the two dimensionless retention times are controlled by two single-loop PID controllers. To feed the MPC with the required internal concentration profiles, a moving horizon estimator (MHE) is developed and added to the control scheme. This new control scheme, MHE + MPC/PID, has been extensively investigated in three case studies. A hypothetical case where the internal concentration profiles are completely available to the MPC, where they come from the MHE processing noiseless measurements, and where they are from MHE processing measurements with added normally distributed random noise. In each of these cases, the control system successfully follows the change in the references over a wide range from complete to incomplete separation, with changes of the product purities from 99 % to 90 % have been investigated. That is due to the use of fully blown chromatographic models used in MHE and MPC, which are solved numerically by the popular mixing cell model. To further improve the computational performance of this control scheme, the space-time solution element/conservation element (CE/SE) method is considered as a numerical tool.

Due to its special formulation, the CE/SE method allows the resolution of steep concentration fronts with only a few dozen discrete elements. In studies of various chromatographic processes, ranging from a simple batch process to a ternary 8-zone centre-cut SMB process with both linear and Langmuir isotherms, it is found to be 10 to 350 times faster than the mixing cell model. Therefore, the CE/SE method is preferred as a numerical method for the proposed control concept.

Finally, the control of SMB processes with the proposed control scheme, for both linear and Langmuir isotherms, has been investigated using the CE/SE method. It is shown that in both cases, i.e. linear and nonlinear adsorption isotherms, reference tracking and disturbance rejection work well if the isotherm parameters are known. In practice, however, isotherm parameters may change due to the adsorbent ageing. As a challenging test scenario, a 10 % step change of the Henry coefficients of the nonlinear Langmuir adsorption isotherms is considered. To handle this scenario, the MHE is augmented with the ability to estimate Henry coefficients online to automatically readjust the operating conditions. It is shown that the extended control system can handle this scenario if the measurement noise is negligible. Lastly, limitations of this approach for highly sensitive processes with steep concentration fronts and measurement noise are also demonstrated for the above challenging test scenario.