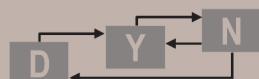


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Band 1/2018

**Jose Roberto Lemoine Nava**

**Online optimal design of experiments and  
parametrization of chemical processes:  
Application to the Simulated Moving  
Bed Process**



# **Online optimal design of experiments and parametrization of chemical processes: Application to the Simulated Moving Bed Process**

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“Insanity: doing something over and over again and expecting a different result.”  
(Albert Einstein)

“I always say the only thing better than finding something you were  
looking for, is finding something you weren’t looking for...”  
(Iroh (fictional character))



## Abstract

The objective of this work is to propose new methods for assisting and improving the parametrization of models of chemical processes using the measurement obtained during their operation. The Simulated Moving Bed (SMB) process has been chosen as a case study. The SMB is a continuous preparative chromatographic separation process. Its performance depends on several factors which affect the adsorption behavior of the chromatographic system, influenced mainly by the characteristics of the stationary phase of the chromatographic columns, but also by operation conditions such as the temperature. The product quality requirements imposed on the SMB process can be met by choosing conservative operating points, at the expense of the economic performance. More optimal operation conditions can be attained by means of model-based operation strategies, provided that the models represent the characteristics of the chromatographic system adequately. The properties of the solid phase in the columns of a SMB plant differ from column to column and change in time due to factors such as aging or damage. However, the measurements from the process can be exploited for tracking the parameters which reflect the changes in the characteristics of the plant.

The first contribution of this work is an optimization-based method tailored for the estimation of the states and the parameters of the individual columns of the SMB plant. The estimation task is difficult, as typically the measurement information available from the process is scarce. The proposed estimation scheme considers explicitly in its formulation the switchings of the sensors at the product ports. The approach shows a good efficiency in tracking some key adsorption isotherm parameters for each individual column of the plant. Additionally, its computational requirements are acceptable and the effort required for its tuning is reasonable.

The second contribution of this thesis is a new approach for the determination of operation conditions to drive chemical processes to deliver measurements which, if used for parameter estimation, lead to estimate parameters with minimum variance. The operation conditions are determined by designing optimal dynamic experiments online, i.e. during the operation of the process, taking into account the most relevant constraints imposed on the process. It is shown that the solution of optimal dynamic experiment design (ODED) problems is very challenging for systems described by models of large dimensions and with many parameters. This is the case of the SMB process if it is intended to estimate all the parameters of the adsorption isotherms for each individual column. An approach for the reduction of the complexity of the online ODED is proposed. The approach consists of decomposing the original experimental design problem into a series of smaller problems which extend a preexisting experiment. Computational strategies for the application of the scheme online are also proposed, and it is shown that they are critical for the success of the method. The approach drastically improves the tractability of the online design of experiments, as well as the computation times needed to solve them. It is shown that in the SMB case study the online solution of the ODED problems is intractable without the proposed approach. Finally, using the proposed method, a strategy for dealing with model-plant mismatch by means of feedback is suggested. Furthermore, the scheme can be used not only for the SMB process, but for any process described by models based on differential equations.

## Kurzfassung

In dieser Arbeit werden neue Methoden vorgeschlagen, die die Parametrierung von Modellen verfahrenstechnischer Systeme während ihres Betriebs unterstützen und verbessern. Mithilfe des sogenannten Simulated Moving Bed (SMB) Prozesses wird die Effektivität der vorgeschlagenen Ansätze beweisen. Der SMB Prozess ist ein kontinuierlicher chromatographischer Prozess. Seine Leistungsfähigkeit ist von zahlreichen Faktoren abhängig, wie die Merkmale der Festphase der chromatographischen Säulen und die Betriebsbedingungen, die das Adsorptionsverhalten des Trennsystems beeinflussen. Die Qualitätsanforderungen an das Produkt können mittels konservativer Prozessfahrweisen erfüllt werden, allerdings zulasten der Wirtschaftlichkeit des Prozesses. Wirtschaftlich günstigere Bedingungen können mit modellbasierten Prozessführungsstrategien erreicht werden, wenn Modelle verfügbar sind, die die Charakteristiken des Trennsystems beschreiben. Jede Säule einer SMB Anlage hat unterschiedliche Merkmale der Festphase, die sich mit der Zeit, z.B. aufgrund von Faktoren wie Alterung oder Beschädigung ändern können. Trotzdem lassen sich die Parameter, die die Veränderungen in der Anlage abbilden, mit Hilfe der aus dem Prozess gewonnenen Messinformationen, bestimmen.

Der erste Beitrag dieser Arbeit besteht aus der Entwicklung eines Ansatzes zur Schätzung der Zustände sowie der Parameter jeder einzelnen Säule der SMB Anlage. Aufgrund der spärlich verfügbaren Messinformation ist die Zustands- und Parameterschätzung im Fall des SMB Prozesses eine große Herausforderung. Dieser Ansatz berücksichtigt ausdrücklich die Umschaltung der Sensoren an den Produktablaufstellen. Mit dem Ansatz ist die zuverlässige Schätzung wichtiger Parameter der Adsorptionsisotherme jeder einzelnen Säule der SMB Anlage möglich. Dabei sind die Rechenanforderungen für die Anwendung des Ansatzes sowie der Aufwand für seine Parametrierung relativ gering.

Der zweite Beitrag dieser Dissertation umfasst einen neuen Ansatz zur Bestimmung von Betriebsbedingungen in verfahrenstechnischen Prozessen, die, unter Zuhilfenahme der aus dem Prozess entstammenden Messinformation, die Schätzung von Parametern mit einer möglichst kleinen Varianz ermöglicht. Die Betriebsbedingungen werden mittels der Planung optimaler dynamischer Versuche während des Prozessbetriebs (Engl. “*online Optimal Dynamic Experiment Design, ODED*”), unter Berücksichtigung der für den Prozess relevanten Nebenbedingungen, bestimmt. Es zeigt sich, dass die Lösung von ODED Problemen für Systeme mit hochdimensionalen Modellen mit zahlreichen Parametern sehr herausfordernd ist. Das ist der Fall, wenn beabsichtigt ist, alle Parameter der Adsorptionsisotherme für jede einzelne Säule beim SMB Prozess zu schätzen. Der Ansatz, um die Komplexität des online ODED Problems zu verringern besteht in der Zerlegung des ursprünglichen ODED Problems in eine Reihe kleinerer Probleme, die einen vorbestehenden Versuch erweitern. Zudem werden rechnerische Strategien, die sich als entscheidend für die erfolgreiche Anwendung der Methode bewiesen haben, entwickelt. Es wird verdeutlicht, dass die ODED Probleme ohne den vorgeschlagenen Ansatz im Fallbeispiel des SMB Prozesses nicht lösbar sind. Schließlich wird für den Umgang mit Modellfehlern eine Feedback-Strategie empfohlen. Darüber hinaus kann der Ansatz nicht nur für den SMB Prozess, sondern vielmehr auch allgemein für alle Prozesse genutzt werden, die sich durch gewöhnliche Differentialgleichungssysteme beschreiben lassen.

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