Journal of Chromatography A, xxx (2016) xxx–xxx

Contents lists available at ScienceDirect

Journal of Chromatography A

journal homepage: www.elsevier.com/locate/chroma

Center-cut separation of intermediately adsorbing target component by 8-zone simulated moving bed chromatography with internal recycle

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ARTICLE INFO

Article history:
Received 25 November 2015
Received in revised form 25 March 2016
Accepted 28 April 2016
Available online xxx

Keywords:
Simulated moving bed chromatography
Center-cut separation
Ternary mixture
Cycloketones
Dead volume

ABSTRACT

An 8-zone simulated moving bed chromatography with internal recycle (8ZSMB-IR) has been designed for center-cut separation, that is, for isolating an intermediately adsorbed component out of a multicomponent mixture. The system consists of two integrated subunits and operates in a fully continuous manner. In the first subunit the feed mixture is split into two fractions containing either a single component or a binary mixture. The binary mixture is recycled through the internal raffinate or extract port into the second subunit, where the target product is isolated. Additionally, the solvent is also recycled internally. For a case study, the separation of a ternary mixture of cycloketones as a model system under weakly non-linear isotherm conditions has been investigated. A few novel configurations of the 8ZSMB-IR unit including the arrangement of the internal recycle of extract, raffinate and solvent streams between two subunits have been examined with respect to various performance indicators for the process realization. The unit performed best with the developed configuration when the internal raffinate stream was recycled and the solvent recycling loop was closed between the last and the first zone of the first subunit. That configuration has further been analyzed experimentally and numerically. On the basis of the results a strategy for determining reliable operating conditions for the 8ZSMB-IR process has been developed. The procedure exploited a model of the process dynamics, which was implemented to refine the isotherm coefficients and to quantify the mixing effect of the liquid stream inside the recycling loops. The upgraded model with the adjusted parameters has been validated based on experimental data and successfully applied for optimizing the operating conditions of the separation.

1. Introduction

Over the past decades, simulated moving bed (SMB) chromatography, has become one of the most powerful and promising techniques for preparative and industrial scale applications. The launch of that technology, which was devised as a practical implementation of continuous true moving bed (TMB), benefits in a significant increase in productivity of the separation process and reduction in solvent consumption compared to batch chromatography [1–4]. The process has been constantly expanding to meet increasing demands in downstream processing.

Many products that are being manufactured by chemical syntheses or biotechnological processes are often obtained along with a number of contaminants, thus advanced purification steps are required to isolate a target compound out of a complex mixture. However, the well-established and robust conventional 4-zone SMB process is limited to split the mixture components only between less and more retained fraction. Therefore, the process has to be modified to accommodate to the separation problem, in which the target product exhibits intermediate elution strength. A great progress has been made to meet that requirement and to develop a technique, which can perform the so-called center-cut separation, i.e., to produce stream of pure product with intermediate affinity to the stationary phase. Several continuous and semicontinuous SMB-based processes have been suggested for realization of that process.

It is worth to start the overview with the JO process, termed also a pseudo-SMB, with ability to perform center-cut separations [3,5,6]. This process operates in semi-continuous mode exploiting batch chromatography followed by the SMB operation without feed delivery. Next, a straightforward approach based on an SMB cas-

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http://dx.doi.org/10.1016/j.chroma.2016.04.083
0021-9673/

cade, which consists of two 4-zone SMB units connected through the raffinate or extract port, has been studied [7–12]. Nevertheless, the latter system is not completely integrated due to different switching time between the coupled units and independent circulation of the mobile phase. Additionally, assembling two SMB units in series involves higher operation and maintenance costs compared to a single SMB unit [13]. Therefore, the concept of a fully integrated 8-zone TMB has been introduced [8], and further theoretically realized by coupling two classical SMB systems into a single device [14–16]. With the passing of time other concepts have been designed, including operations with five zones, e.g., side-stream SMB [17], 5-zone SMB process [13,18–21], and three-fraction SMB (3F-SMB) [22]. For separation of mixtures with a high separation factor between the intermediately adsorbed component and more retained impurities 2-zone SMB/Chromatography system was proposed [23]. Moreover, new SMB-based techniques have been developed to separate the multicomponent mixtures of proteins, such as Multicolumn Countercurrent Solvent Gradient Purification (MCSGP) process, where continuous countercurrent SMB process is realized in the solvent gradient mode [24,25], and gradient with steady state recycle (GSSR), where a multicolumn open-loop system is used to perform solvent gradient separation [26]. Further, Intermittent-SMB (I-SMB) for separating multicomponent mixtures into three fractions has been designed based on configuration of four sections along a column train [27], and later by three column in cascade operation [28], where the switching time in both cases is divided into two substeps differing with flows [27,28]. Also, Multi-column Recycling Chromatography (MCRC) method has been suggested for chromatographic fractionation of multicomponent mixtures by implementing four-column technique, which operates in four steps with closed loop recycling to provide an intermediately eluting compound [29,30]. Another interesting but more complicated approach to perform ternary separation was proposed as Generalized Fully Cycle (GFC) formulation, where each of the four steps can be operated in a distinct manner based on various SMB alternatives [16,31]. Recently, the potential of GFC strategy has been exceeded by Full Superstructure including all possible locations of the inlet and outlet streams to find the optimal operation scheme [32].

In the present study, the integrated 8-zone simulated moving bed chromatography with internal recycle (8ZSMB-IR) has been investigated to perform center-cut separation with high productivity and yield. The 8ZSMB-IR unit operates in a fully continuous manner and enables efficient center-cut separation of multicomponent mixtures with a low separation factor. The system is characterized by relatively simply design and is robust thanks to the eight sections applied. Moreover, the integration of two SMB subunits in a single system has a potential to improve separation productivity and reduce solvent consumption compared to other techniques, which may compensate difficulties with operating such a complex system [8,14–16]. Though the 8ZSMB-IR process has been theoretically studied in several publications, the only experimental validation was performed by Nowak et al. [33]. Because that concept has not yet been fully understood, this study is aimed at a detailed analysis of its effectiveness. The presented work extends previous studies and provides an efficient procedure for the process realization, including: investigation of the various unit configurations, followed by construction of a dynamic model for determination of reliable operating conditions and process prediction. At first, experimental data acquired in a batch separation system were exploited to formulate the dynamic model and determine underlying model parameters. Then, various configurations of the 8ZSMB-IR unit have been numerically analyzed, including the arrangement of internal recycling of the extract and raffinate stream. It has been shown that the process performance can be improved by a proper choice of the position of the solvent recycling loop and desorbent delivery. Finally, the separation has been realized experimentally for the most efficient configuration selected according to the results of the model simulations. On the basis of the experimental concentration profiles recorded at the 8ZSMB-IR outlet ports, the model has been improved by adjusting the isotherm coefficients and accounting for the mixing effect within the internal recycling loops. The upgraded model was capable to predict the process performance for various operating conditions.

2. Theory of 8ZSMB-IR chromatography

2.1. Process concept

The idea of the 8ZSMB-IR process can be described with the reference to a simplified flowsheet scheme based on the extended approach to the classical SMB concept, which is illustrated in Fig. 1. The system is fully continuous and integrated; it operates with the same switching time, t_s, for all ports at continuous circulation of the mobile phase. It consists of eight zones connected in a series by eight flow-through ports. Each zone may contain one or more fixed-bed columns. Three inlet streams are delivered into the unit: feed (F), desorbent-I (D-I), desorbent-II (D-II). Three outlet streams leave the system: raffinate-II (R-II), extract-II (E-II) and raffinate-I (R-I) or extract-I (E-I), depending on the unit configuration. Accordingly, internal recycle (REC) of either raffinate-I or extract-I can be applied.

Each zone accomplishes specific function during the separation process. Generally, two types of zones can be distinguished, i.e., separation and regeneration zones. In zones II, III, VI, VII the separation takes place, while zones I and V are used to regenerate the solid phase by desorbing more adsorbed components into the extract streams, and zones IV and VIII for recovering the liquid phase by removing less retained components into the raffinate streams. To accomplish the solid phase regeneration, fresh desorbent is continuously pumped at the inlet of zones I and V. In analogy to the classical 4-zone SMB, the countercurrent movement between the solid and liquid phases is simulated by a periodic shift of all inlet and outlet ports in the direction of the fluid flow. Hence, each column changes its position simultaneously by one to the back after a defined switching time interval. When each column passes all eight zones, a full cycle is completed.

The feed stream is delivered between the central separation zones II and III. It contains an early eluting component A, a component B with intermediate adsorption strength and a strongly retained component C. The components A and C may be assumed to be impurities that consist of more than one ingredient. Due to different adsorption affinities to the stationary phase, the concentration profiles migrate through the columns with different velocities, and the less retained components are carried with the liquid phase to the raffinate ports, while the more retained ones are transported upwards with the solid phase to the extract ports. After initial period, which takes several cycles, the process approaches a cyclic steady state (CSS) with periodically repeating concentration profiles inside the columns. To isolate the target component B, the ternary mixture is pre-separated in the subunit-I of the system, where either raffinate-I containing component B and less retained component A (configurations ***a–c*** in Fig. 1A–C) or extract-I containing component B and more adsorbed component C (configurations ***d–f*** in Fig. 1D–F), is internally recycled. The internal stream is recycled into a port between the separation zones VI and VII of subunit-II to accomplish the separation. In such a way the pure target B is collected in the port of extract-II for the configurations ***a–c*** or raffinate-II for the configurations ***d–f***.

For both options of internal recycle of either raffinate-I or extract-I stream, the solvent can also be recycled by closing the liq-