SINTHESIS OF 1,1-DIBUTOXYETHANE IN A SIMULATED MOVING-BED ADSORPTIVE REACTOR.

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Summary

The synthesis of 1,1-Dibutoxyethane was carried out in a Simulated Moving Bed pilot unit LICOSEP 12-26 (Novasep, France) with 12 columns packed with the commercial ion-exchange resin Amberlyst 15 (Rohm & Haas, France). Experimentally it was obtained a raffinate purity of 85% with a productivity of 5,04 $Kg.L^{-1}.d^{-1}$ and a desorbent consumption of 15, 5 $L.Kg^{-1}$. The influence of the different operation parameters was studied by simulation. The results showed that the SMBR process presents a potential to be a competitive, efficient and environmentally friendly way to produce 1,1- Dibutoxyethane and other acetals.

Keywords

Multifunctional reactors, simulated moving bed, acetals, amberlyst 15

Introduction

Acetalization reactions carried out in conventional batch reactor present low conversions, due to the equilibrium limitations [1]. The use of integrated reaction/separation processes appears to be a good alternative in order to enhance the reaction conversion, since they allow the separation of the products from the reaction medium as they are formed, displacing the chemical equilibrium towards product formation [2]. Among the integrated reaction/separation processes reactive chromatography is a very attractive way to increase the reactants conversion [3] and in terms of energy consumption savings, since is based on the selective adsorption rather selective evaporation; therefore, the use of high temperatures is not necessary.

The aim of the present work is the study of the performance and feasibility of the synthesis of 1,1 –Dibutoxyethane in a simulated moving bed reactor. The SMBR model is validated by comparison with experimental data. The applicability of TMBR model in the simulation of SMBR operation is verified. The effect of SMBR parameters in the process performance is studied by simulation.

Experimental Section

The SMBR experiments were carried out in a pilot unit Licosep® 12-26 by Novasep. It is a continuous chromatographic system constituted by 12 columns connected in series, packed with Amberlyst®15 (Rohm and Haas). The internal concentration profiles are determined by collecting samples by a six-port valve located between the twelfth and the first column.

All the experiments were carried out with a SMBR configuration of 3 columns per section (3-3-3-3), flowrate of 45 mL/min in section 1, and at 25ºC. The feed composition was a mixture of 1-butanol and acetaldehyde, with 50% acetaldehyde molar fraction.

Figure 1 shows the experimental and simulated cycle steady-state concentrations profiles in the SMBR unity for the best raffinate purity performance.

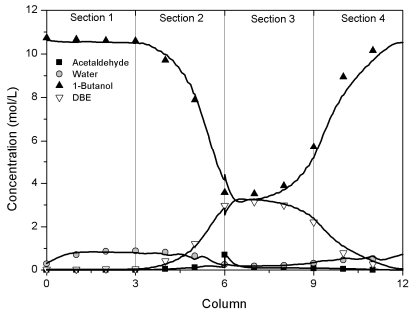

Figure 1. Experimental and simulated (by SMBR model) cycle steady-state concentration profiles.

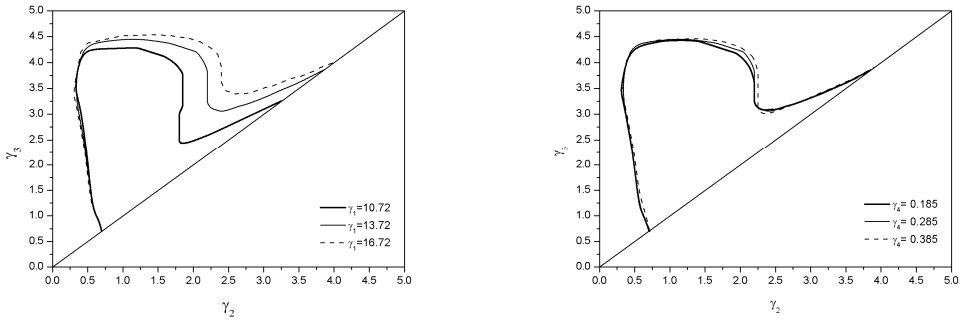

Figure 2. Effect γ_1 and γ_4 on reaction/separation regions for a minimum value of extract and raffinate purities of 95%.

Mathematical Model

The concept of True Moving Bed Reactor (TMBR) can be used as an alternative in order to predict the SMBR process operation. The equivalence between TMBR and SMBR models is made by keeping constant the liquid velocity relative to solid velocity.

Figure 3 presents a comparison between the steady-state internal concentration profiles given by the TMBR model and the SMBR model.

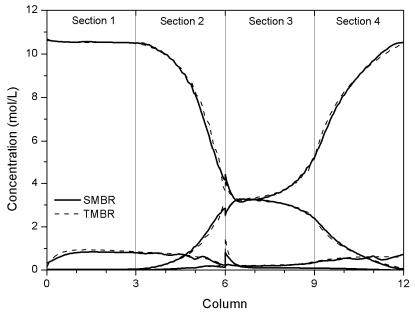

Figure 3. Comparison of the SMBR cycle steady-state concentration profiles calculated by TMBR and SMBR models

Reactive/separation regions

The correct choice of the flowrate in each section is crucial for the successful operation of the SMBR chromatographic reactor. For each section j the parameter γ_j represents the ratio between the fluid interstitial velocity, u_j, and the solid interstitial velocity, u_s. The reaction/separation region defines the operation region in the γ_2-γ_3 plane where a minimum value of extract and raffinate purity is guaranteed. In the reaction/separation regions construction the constraints relatively to γ_1 and γ_4 should be met ($\gamma_{1,min}$=6.866 and $\gamma_{4,max}$ =0.570)

Figure 2 shows the influence of γ_1 and γ_4 on the reaction/separation regions.

Conclusions

The synthesis of 1,1- Dibutoxyethane was carried out by reacting 1-butanol and acetaldehyde, using Amberlyst 15 as catalyst/adsorbent, in a simulated moving bed reactor, it was obtained a minimum conversion of 96% and the best raffinate purity of 85.1 %. The comparison between experimental and simulated data shows that both SMBR and TMBR provide a good representation of SMBR operation. The effect of different SMBR parameters on the SMBR performance and on the reaction/separation regions was studied. The size of reaction/separation regions increases by increasing both γ_1 and switching time; however, this increase leads to a worse performance in terms of productivity and desorbent consumption.

References

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