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# Coupled PermSMBR – Process design and development for 1,1-dibutoxyethane production<sup>☆</sup>

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## ABSTRACT

In this work, a new configuration of the simulated moving bed membrane reactor (PermSMBR) technology is presented, the coupled PermSMBR, where the tubular membranes are located after fixed-bed columns packed with the catalyst/adsorbent. By this way the membranes are not in contact with the solid, which from an industrial point of view is easier to implement since the process of membranes installation/replacement and clean-up is simpler than in the previous considered set-up (tubular membranes packed with the catalyst/adsorbent – integrated PermSMBR). The 1,1-dibutoxyethane production is used, as an example, and the features of the new “coupled PermSMBR” and the previous set-up (integrated PermSMBR) are discussed. The coupled PermSMBR revealed to be a very attractive solution for the sustainable 1,1-dibutoxyethane production, proved by the high productivity and low desorbent consumption obtained within the studied conditions.

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**Keywords:** Process intensification; Membrane reactors; Simulated moving bed reactor; Hybrid technologies; 1,1-Dibutoxyethane; Silica membranes; Amberlyst-15

## 1. Introduction

The simulated moving bed membrane reactor (PermSMBR) is a recently developed technology that consists of the integration of permselective membranes with the simulated moving bed reactor (SMBR) (Silva et al., 2009). The SMBR is implemented in well-known, but nontrivial, SMB equipment (Broughton and Gerhold, 1961), where the columns are packed with a solid catalyst with adsorptive properties or with a mixture of solid catalyst and adsorbent particles. The standard SMBR configuration comprises two inlet streams (feed and desorbent) and two outlet streams (extract and raffinate) and the countercurrent solid movement is simulated by a synchronous shift of these streams by one column in the direction of the fluid, at regular time intervals called the switching time. If the feed comprises two reactants (A and B), in which, for instance A, is used as desorbent, and A and B react to give two products, C and D, the latter being

more adsorbed than the former, then a mixture of D and A is obtained in the extract and a mixture of C and A in the raffinate.

The inlet/outlet streams divide the unit into four different sections, each one with a specific role (described in a previous work (Rodrigues et al., 2012)) and having a given number of columns (see Fig. 1). This is the SMBR principle of operation which is similar to the one of the PermSMBR. However, in the new equipment each column is replaced by a set of tubular membranes packed with the solid (catalyst with adsorptive properties) or the mixture of solids (catalyst and adsorbent) (Fig. 2). Besides, another stream is collected: the permeate stream that combines all the flows removed through the membrane; the permeate is therefore rich in the product for which the membranes are selective. Depending on the system, different membrane processes can be applied as microfiltration, ultrafiltration, vapour permeation, pervaporation, among others.

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**Nomenclature**

$a_i$	liquid-phase activity of component $i$ in bulk side
$A_m$	membrane area per unit reactor volume ( $\text{m}^2_{\text{membrane}}/\text{m}^3_{\text{bulk}}$ )
$C$	liquid phase concentration ( $\text{mol}/\text{m}^3$ )
$\bar{C}_p$	average liquid phase concentration inside the particle ( $\text{mol}/\text{m}^3$ )
$D_{ax}$	axial dispersion coefficient ( $\text{m}^2/\text{min}$ )
$DC$	desorbent consumption ( $\text{m}^3/\text{mol}$ )
$d_{int}$	membrane internal diameter (m)
$J_i$	permeate flux of species $i$ ( $\text{mol}/(\text{m}^2\text{min})$ )
$k_{bl}$	boundary layer mass transfer coefficient (m/s)
$k_e$	external mass transfer coefficient (m/s)
$k_i$	internal mass transfer coefficient (m/s)
$K_L$	global mass transfer coefficient (m/s)
$k_{ov}$	global membrane mass transfer coefficient ( $\text{mol}/(\text{m}^2 \text{ s Pa})$ )
$L$	column length (m)
$L_m$	membrane length (m)
$n$	total number of components
$N_c$	total number of columns
$p_i^0$	saturation pressure of component $i$ (bar)
$P_{perm}$	total pressure on the permeate side (bar)
$PR$	raffinate productivity ( $\text{kg}_C/(\text{L}_{resin} \text{ day})$ )
$PUR$	raffinate purity (%)
$PUX$	extract purity (%)
$q$	solid phase concentration in equilibrium with the fluid concentration inside the particle ( $\text{mol}/\text{L}$ )
$Q$	volumetric flow rate ( $\text{L}/\text{min}$ )
$Q_{memb}$	permeance ( $\text{mol min}^{-1} \text{ m}^{-2} \text{ Pa}^{-1}$ )
$r$	rate of reaction ( $\text{mol kg}^{-1} \text{ min}^{-1}$ )
$Re$	Reynolds number
$Re_p$	Reynolds number relative to particle
$r_p$	particle radius (m)
$Sc$	Schmidt number
$Sh$	Sherwood number
$t$	time variable (min)
$t^*$	switching time (min)
$u$	interstitial velocity (m/min)
$v$	superficial velocity (m/min)
$V_C$	volume of packed column ( $\text{m}^3$ )
$V_{mol,i}$	molar volume of species $i$ ( $\text{L}/\text{mol}$ )
$X$	acetaldehyde conversion
$y_i$	molar fraction in the vapour phase of component $i$
$z$	axial coordinate (m)

**Greek letters**

$\gamma^*$	activity coefficient
$\varepsilon$	bulk porosity
$\varepsilon_p$	particle porosity
$\nu_i$	stoichiometric coefficient of component $i$
$\rho_b$	bulk density ( $\text{kg}/\text{m}^3$ )
$\rho_p$	particle density ( $\text{kg}/\text{m}^3$ )
$\mu$	viscosity (cP)
$\eta$	effectiveness factor of the catalyst
$\Delta P$	pressure drop (Pa)
$\Phi$	sphericity of the particles

**Subscripts**

$i$	relative to component $i$ ( $i = A, B, C, D$ )
$j$	relative to section ( $j = 1, 2, 3, 4$ )
$k$	relative to column in coupled PermSMBR
$m$	relative to membrane module in coupled PermSMBR
$O$	relative to initial conditions
$A$	relative to n-butanol
$B$	relative to acetaldehyde
$C$	relative to 1,1-dibutoxyethane
$D$	relative to water
$F$	relative to the feed
$p$	relative to the particle
$R$	relative to raffinate
$Rec$	relative to recycle
$X$	relative to extract

The PermSMBR concept was already evaluated for the production of acetals (1,1-diethoxyethane and 1,1-dibutoxyethane) and a green solvent (ethyl lactate), using Amberlyst-15 wet (A15) resin as catalyst/adsorbent and silica membranes (from Pervatech BV, the Netherlands) for water dehydration (by product in both acetalization and esterification reactions) (Silva et al., 2010; Pereira et al., 2012). The results showed a productivity enhancement accompanied by a significant reduction on the solvent consumption when compared with reactive distillation and/or the SMBR technology, proving this equipment high potential for the production of oxygenated compounds.

In this work, a new PermSMBR configuration is proposed, where each SMBR column is followed by a membrane module – the coupled PermSMBR (see Fig. 3). In the coupled PermSMBR the membranes will not be in contact with the catalyst/adsorbent, which from an industrial point of view will be easier to implement since the process of membranes installation/replacement and clean-up is simpler. Moreover, the mechanical and chemical resistances of membranes will not be compromised by the catalyst/adsorbent presence. The assessment of this new set-up is made using, as an example, the production of 1,1-dibutoxyethane (DBE) and comparing its performance with the one of the SMBR and integrated PermSMBR under different operating conditions.

DBE was selected since it can be produced from common bio-refinery building blocks (n-butanol and acetaldehyde) according to Scheme 1, and used as green fuel additive enhancing the renewable fraction in diesel, which positively contributes for the important European environmental commitment that aims to replace 10% of total transport fuels by biofuels by 2020 (UE Directive 2009/28/EC). Moreover, DBE diesel blends have higher cetane numbers than diesel (Boennhoff and Obenaus, 1980).

## 2. Mathematical model of the coupled PermSMBR

As for the previous studied PermSMBR configuration that will be called integrated PermSMBR, the mathematical model used



**Scheme 1**

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