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A process integration approach for the production of biological iso-propanol, butanol and ethanol using gas stripping and adsorption as recovery methods



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ABSTRACT

Biomass fermentation to Iso-propanol, Butanol and Ethanol (IBE) is particularly important as IBE is a common building block in the development of biorefineries and IBE-producing bacteria are robust industrial organisms, capable to utilize the sugars of the lignocellulosic biomass. Research is focused on increasing fermentation yields and the reduction of energy that is required to separate the volatile products. The paper addresses both of these challenges combining experimental innovations with a systems engineering approach. IBE is recovered from a gas-stripped fermenter whose potential for adsorption is researched and integrated with downstream options for separation. Design and integration is assisted using a systems approach that relies on mathematical models that regress and extrapolate experimental data for scale-up calculations. Process integration involves synthesis challenges to define biorefinery portfolios and systems integration to combine fermentation, stripping, adsorption, and distillation. The analysis considers 4 alternative biorefinery cases and presents results with significant savings in energy use and costs (up to 87% savings reported) after the application of energy integration to the IBE plant. Scenarios are analysed economically and confirm benefits in the use of adsorption and viable production yields.

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Abbreviations: ABE, Acetone, Butanol, Ethanol; IBE, Iso-propanol, Butanol, Ethanol; ISPR, “in-situ” product removal; i, adsorbable components including iso-propanol (I or IPA), butanol (B or BUT), ethanol (E or ETOH), water (W or WAT); x,t,u, bed axial distance [m], time [sec] and gas mixture velocity [m/sec]; ε ,L, void fraction [–] and length [m] of column bed; C_i , C_i^e and C_i^f , concentration of component i in the void space, the void space at equilibrium and at the feed of adsorption column [g_i/m³]; q_i , q_i^e and q_i^0 , actual, at equilibrium and maximum adsorbed capacity of component i per gram of adsorbent [g_i/g_s]; k_{bi} , Langmuir constant of component i [m³/g_i]; D_{bi} , k_{fi} , axial dispersion coefficient [m²/sec] and mass transfer coefficient [m/sec] of component i; ρ_s , d_p and R_p , bulk density [kg_s/m³], particle diameter [m] and radius [m] of adsorbent; C_i^l , K_s , liquid concentration inside reactor [g_i/l] and stripping rate [1/h] of component i; F, B, D, feed, bottom and distillate flowrates of condenser flask [mol/h]; z_i , x_i , y_i , feed, bottom and distillate molar compositions of condenser flask of component i; P_i^s and P, saturated vapor pressure [bar] of component i and vessel pressure [bar]; k_i and γ_i , volatility [–] and activity coefficient [–] of component i; ψ , distillate (D) to feed (F) ratio; r_{di} , k_{di} , k_{di}^0 , ΔH_i and n_i^d , reaction rate of desorption, desorption constant [1/sec], pre-exponential factor [1/sec], heat of desorption [kJ/mol] and order of desorption [–] of component i; R, T, gas constant [kJ/mol/K] and temperature [K].

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1. Introduction

Biorefineries are backbone industries of the emerging bio-based economy. Biorefineries co-produce biofuels and chemicals using a wide variety of feedstocks that include lignocellulosic biomass, oleo-chemicals, organic waste or algae. Second-generation plants are refrained from any competition with food supplies focusing primarily on forestry, wood residues, organic waste and agricultural by-products [1,2]. Lignocellulosic biomass is the predominant supply and its interim conversion to sugar streams offers numerous opportunities for exploitation. Accordingly, lignocellulosic biorefineries are more advanced and closer to commercial applications. Their value chains are vast and extensive but some of these paths are well developed and very promising [3]. One of the most promising involves the fermentation of sugars to acetone, n-butanol and ethanol (ABE) and/or iso-propanol, n-butanol and ethanol (IBE). Production routes to ABE and IBE have both drawn significant attention as they involve common biomass blocks from different types of supplies and stand capable to build the required capacities for industrial plants [4].

The improvement of yields is challenged by the integration of reaction and separation that each, separately, involves low yields

and a heavy use of energy [5]. The low concentrations in ABE and/or IBE (typically 2% v/v) are primarily attributed to the toxicity of products (mainly butanol) in the strains. The volatile products are recovered using distillation and fractionation, both expensive due to the diluted streams from the fermenter. Due to the low concentrations, alternative separation methods to distillation are attractive and promising to integrate; they include options for extraction, adsorption, gas-stripping and membrane separation (including pervaporation and perstraction) [5–12].

Oudshoorn et al. [10] has compared options in separating 1-butanol from aqueous solutions. Oudshoorn et al. concluded that adsorption (basis 8 MJ/kg butanol) makes the most efficient choice and a superior option over distillation (29.6 MJ/kg butanol). Several adsorbents have been tested to this purpose. Studies [13] report the use of activated carbon F-400 over options that include activated carbon F-600, zeolites (NaY and ZSM-5) and silicalite resins (HiSiv 3000). Gas-stripping process is another option to capture and separate volatile chemicals [14]. The process is relatively simple, brings no harm to the culture and is relatively easy to function in continuous mode [15,16]. Gas is sparged through a medium that captures the volatile products. The products are further condensed, removed and separated from each other. Experimental studies invariably report higher process yields and improvements in the utilization of the sugar substrate [9,16–19]. To address the impact of toxicity in production, *in-situ* product removal (ISPR) methods are proposed to remove the products during fermentation. ISPR refrains from inhibition and enhances the utilization of substrates. *In-situ* removal yields concentrated feed solutions reducing throughput and, subsequently, equipment size and cost. ISPR has been recently reviewed by Van Hecke and co-workers [20].

Scale-up costs are challenged by the large fermentation volumes and the separation costs to recover and purify alcohols. The integration of reaction and separation can apparently reduce costs and improve efficiencies; however, options to integrate differ significantly. A systems approach is able to review schemes and explain trade-offs. Several models are already reported in the literature for the individual recovery of chemicals. The models include extraction [21], condensation [22] and gas stripping [23–25]. More rigorous models (CFD) are also available to study fermenters [26]. Shortcut models are apparently useful to screen designs and estimate trade-offs between capital and operating costs [27]. Staggs and Nielsen [28] and Stoffers et al. [29] have studied such alternatives for the recovery of butanol. A wide range of recovery and purification options are presented by Kujawska et al. [30]. Several flowsheeting studies are also available in the literature [31,32]. Designs of industrial and commercial scale (150 ktn/yr of ABE) are presented by Qureshi and Blaschek [33]; they incorporate distillation, stripping and membranes. Kumar et al. [34] compared designs that include pretreatment. Superstructure methods have been applied to address sequencing of separations and integration [35].

This work presents a systems approach that combines experimental work with process modeling to develop integrated designs for the production of IBE. The fermentation process is integrated with the recovery and purification of iso-propanol, butanol and ethanol using process synthesis technologies to review trade-offs. The approach reviews options for *in-situ* separation that involves gas-stripping and adsorption with activated carbon; condensation was also examined for the efficient recovery of stripped alcohols instead of adsorption. Distillation coupled with decantation is deployed to bypass alcohol-water azeotropes and provide high purity alcohol products. The analysis addresses the development of product portfolios, the selection of recovery-purification technologies, process operation specifications and accordingly reports recovery yields for the overall process. Results are presented for 4 biorefinery cases and include product yields, energy efficiencies and approximate estimates for the capital and operating cost.

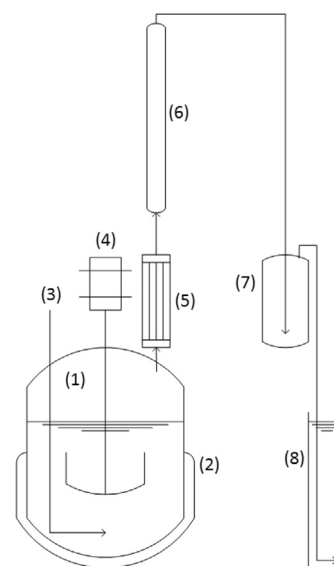


Fig. 1. Set-up used for experiments: (1) Reactor (of 0.5 L and of 2 L), (2) Heating jacket, (3) N₂ stripping gas, (4) Agitator, (5) Vertical condenser (10 °C), (6) Adsorption column (packed with Sorbonorit® B3), (7) Condenser flask (–5 °C) and (8) wash water (0 °C).

2. Materials and methods

The analysis makes use of data produced from an experimental rig that is described with respect to the strains and its cultivation media, the fermentation process and the scheme deployed for N₂ stripping and adsorption. The scheme is shown in Fig. 1 and is explained in the following sections.

2.1. Experimental methods

2.1.1. Strains and cultivation media

The laboratory strain *C. beijerinckii* NRRL B593 is stored, as spore suspension, at 15% (v/v) glycerol and at –20 °C. Prior to the inoculation of pre-cultures, the suspension is heat-shocked in a water bath for 5 min at 80 °C. The culture medium is made anaerobic by sparging with nitrogen gas following previous work [36]. Cultures and pre-cultures are produced using a modified CM1 medium that contains (per liter): yeast extract: 2.5 g; KH₂PO₄: 1.0 g; K₂HPO₄: 0.76 g; ammonium acetate: 2.9 g; *p*-amino-benzoic acid: 0.10 g; MgSO₄·7H₂O: 0.4 g; glucose: 60 g; and FeSO₄·7H₂O: 0.1 g. Excessive foaming is prevented using antifoam Sigma A204.

2.1.2. Adsorption of IBE and water: single-component gas phase equilibrium

The adsorption experiments use activated carbon (Sorbonorit® B33), courtesy of Cabot Norit Nederland B.V. The adsorbent is dried at 75 °C and is stored at room temperature. The gas phase equilibrium experiments are performed in closed desiccators (previously dried with silica). Approximately 1 g of adsorbent is used per 20 g of butanol, iso-propanol, ethanol or water. Each component was incubated at room temperature and the adsorption time was 72 h; the process follows the steps suggested in [37]. Each test is carried out at least in triplicate.

2.1.3. Gas stripping of model solutions with and without adsorption

The experiments use the set-up shown in Fig. 1. The solutions of iso-propanol and butanol are prepared in demineralized water at concentrations 6 and 10 g/L respectively. Solutions of 0.5 L are placed in 0.75 L bioreactors (Multifors 2, Infors HT) and are sparged

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