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Adaptable dividing-wall column design for intensified purification of butanediols after fermentation

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ABSTRACT

The 2,3-, 1,4- and 1,3-butanediols (BDOs) are valuable platform chemicals traditionally produced through petrochemical routes. Alternatively, there is growing interest in synthesizing these chemicals through fermentation processes. However, several drawbacks of the fermentation process (e.g. low product concentration, formation of by-products and high-boiling temperatures of BDOs) hinder the downstream process and increase overall production costs. This original research proposes an advanced large-scale (processing capacity of 160 ktonne/y) process design for the purification of different BDOs after fermentation. The initial preconcentration step removes most water and light impurities in heat pump-assisted distillation column. The heart of the developed process is an integrated dividing-wall column that effectively separates high-purity BDO (>99.4 wt% in all cases) from the remaining impurities. Each BDO isomer was purified cost-effectively (0.208 – 0.243 $/kg_{BDO}$) and energy-efficiently (1.854 – 2.176 $kW_{th}h/kg_{BDO}$) using a single process design, offering flexibility in the development of sustainable bioprocesses for BDO production.

Keywords: butanediols, dividing-wall column, downstream processing

INTRODUCTION

Uncertainties related to fossil fuel-based production pathways are driving interest in developing sustainable alternatives. Fermentation emerges as a promising substitute for the petrochemical production of many chemicals. Among these potential bioproducts, butanediols (BDOs) are very important platform chemicals with a wide variety of applications (e.g. pharmaceuticals, cosmetics, softening agents, solvents, plasticizers, fertilizers, printing inks, polymers, etc.). Significant research effort has been put into developing genetically engineered microorganisms that can produce BDOs from different renewable carbon sources. So far, fermentative production of 2,3-, 1,4- and 1,3-butanediol resulted in significant product titers and may have potential for industrial-scale production. Contrarily, titers of 1,2-BDO achieved so far are very low and additional improvements in the fermentation are needed before considering scale-up. Despite the significant research on developing the upstream process, the recovery of BDOs from fermentation broth has not been nearly as promptly addressed. Given the drawbacks of the fermentation process (low product concentrations, formation of by-products, high boiling temperatures of BDOs, etc.), the costs of the separation process may significantly contribute to the total production costs.

PROBLEM STATEMENT

Published studies on BDO recovery after fermentation mainly focus on 2,3-BDO, with comparatively less research dedicated to the recovery of other BDOs. Due to the complex composition of the fermentation broth and high boiling point of BDO products, several steps are required in downstream processing. Consequently, costs of the purification process may significantly contribute to the total production costs [1]. Different techniques have been considered for the separation of BDO from fermentation broth (e.g. solvent extraction, salting-out extraction, sugaring-out extraction, reactive extraction, pervaporation, etc.) [1–5]. Nonetheless, these methods have some drawbacks that hinder effective large-scale implementation (low product recovery, large amounts of required solvent, subsequent solvent recovery, large amounts of salting-out agents, potential solvent toxicity, significant waste production, membrane fouling, etc.). Moreover, these techniques require additional steps to obtain high-purity BDO product. Alternatively, conventional filtration and ion exchange steps can be used to remove most of the microorganisms, large organic molecules and salts (see Figure 1, preconcentration and final purification (green boxes) are the focus of this research). After these initial downstream processing steps, additional purification is needed to remove the remaining water and obtain a high-purity BDO product. Due to the significant temperature differences and absence of azeotropes, distillation may be used for both preconcentration and final purification. Nonetheless, distillation implies evaporating large amounts of water and advanced energy-saving techniques are required to make the process efficient. Several distillation-based configurations have been proposed (consecutive distillation, extractive distillation, multi-effect distillation, dividing-wall column distillation) [6–9] with energy requirements of 3.1 – 6.5 $kW_{th}h/kg_{BDO}$ (~6.2 to recover 90% of 2,3-BDO using dividing-wall column [10]).

The main goal of our research is to significantly decrease the energy requirements of the preconcentration and final purification parts of the recovery process. Recently, we have achieved this goal by developing a state-of-the-art large-scale downstream processing design (broth processing capacity of 160 ktonne/y with a production capacity of 11 – 15 ktonne/y) that may be easily adapted to purify 2,3- (case 2-3), 1,4- (case 1-4) or 1,3-BDO (case 1-3) after fermentation, and conventional filtration and ion-exchange steps [11]. These initial filtration and ion exchange steps are commonly used in industrial-scale purification processes and were not designed in this study.

METHODS

Aspen Plus was employed as a computer-aided process engineering (CAPE) tool to design BDO purification processes, whereby rigorous simulations were performed for all process operations. The compositions of the fermentation broths from published literature [7, 12, 13] were used to obtain feed streams for the recovery process (see Figure 3). Generally, concentrations of BDO and water are about 7 – 9 wt% and 87 – 91 wt%, while some light impurities (ethanol, formic acid, acetic acid, etc.) and heavy impurities (lactic acid, succinic acid, glucose, etc.) are present in all cases. Thermodynamic interactions between different components in the broth were described using the Non-Random Two Liquid (NRTL) property model. Hayden-O'Connell (HOC) extension was used to describe vapor phase interactions if polar components (e.g. carboxylic acids) are present (cases 2-3 and 1-3)[14].

RESULTS AND DISCUSSION

This section contains the main results related to the design of large-scale processes for the recovery of BDOs after fermentation. Flowsheets for these cases, with the main elements of mass and energy balances, are presented in Figure 3.

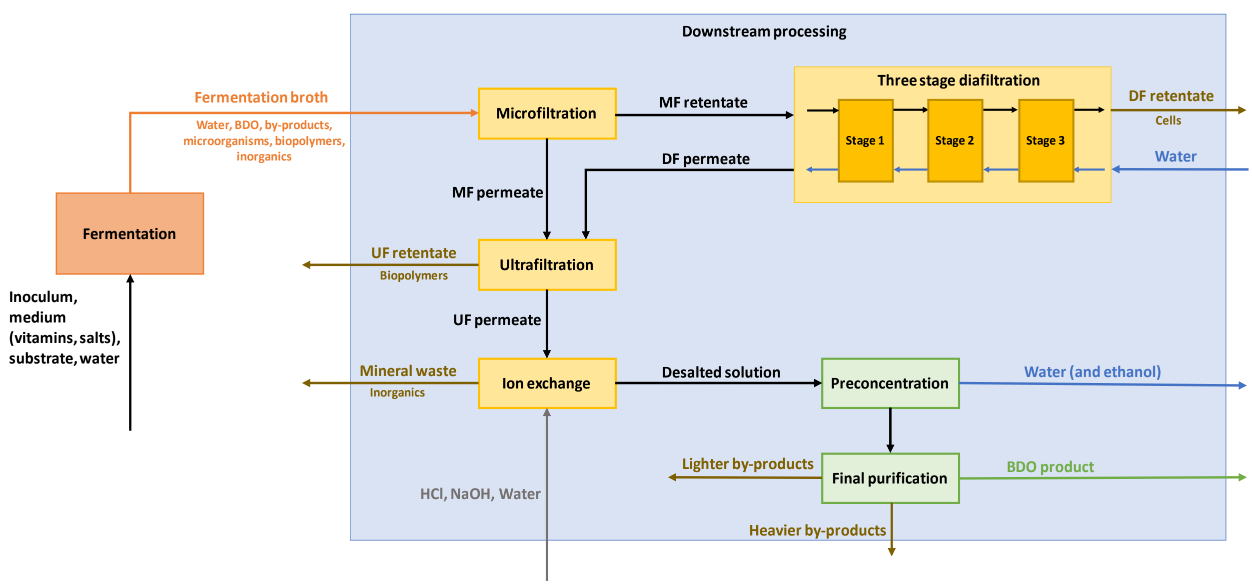

Figure 1. Block flow diagram of the complete BDO production process

Preconcentration step

Following the initial filtration and ion exchange steps necessary to remove microorganisms, large organic molecules and inorganics, the fermentation broth is still very dilute. Hence, a preconcentration step is performed in distillation column C1 to separate most of the water with some light impurities (distillate), from BDO with heavy and remaining light impurities (bottom product). Reduced pressure operation (top pressure of 0.130 bar) was chosen to avoid high temperatures, facilitate separation and decrease energy requirements. Due to the applied vacuum, structured packing was assumed for this column's internals. Given the high concentration of water in broth, the preconcentration step is very energy intensive (> 12 MW_{th}). Thus, the implementation of advanced energy-saving techniques is crucial for the energy efficiency of the whole recovery process. Theoretically, all components lighter than BDO can be separated as the top product of column C1. However, this would limit the application of heat pump systems as the temperature difference between the top and the bottom of column C1 would be very large. On the contrary, the proper choice of operating parameters (distillate-to-feed ratio) allows the implementation of a mechanical vapor recompression system (MVR) [15]. This heat pump system implies compressing vapor from the top of a column and using it as a heating utility in a reboiler. The measure of obtained energy savings by implementing this system is the coefficient of performance (COP) – the ratio of exchanged thermal energy and required compressor power. COP values of the MVR systems implemented to column C1 are > 14 in all cases. Given that a COP of over 2.5 testifies to the energy efficiency of heat pump systems, it may be concluded these MVR systems result in significant energy savings. Valorisation of by-products from the top aqueous stream of column C1 was not considered due to their low concentration. Instead, as the worst-case scenario, it was assumed that this stream would be sent to wastewater treatment and appropriate costs were accounted for.

The benefits of the proposed multi-stage preconcentration step in the heat pump-assisted distillation column are manifold. Firstly, removing most of the water drastically reduces equipment size and reboiler duty for the final purification step. Secondly, multi-stage preconcentration in the distillation column allows the separation of water and light impurities without losing BDO product. Thirdly, the applied MVR system significantly decreases energy requirements for the overall recovery process and enables complete (green)electrification of this step. To our knowledge, a multi-stage preconcentration step that removes most of the water and light impurities without losing BDO product and that can be powered completely by electricity has not been proposed so far.

Further purification

Following the preconcentration step, additional processing is needed to obtain a high-purity final BDO product. This may be performed in a sequence of two distillation columns, whereby the first one removes the remaining water and light impurities, while the second one separates BDO from heavy impurities. Alternatively, these two columns may be integrated into one dividing-wall column (DWC) with a divided overhead section and common bottom section [16]. This highly integrated system combines two columns into one shell with two condensers and only one reboiler. In addition to reducing capital costs (through the number of columns, heat exchangers, and required area for installation), DWC may significantly reduce energy requirements for separation. Although DWC technology has certain limitations, such as a potentially more complex control strategy, its implementation has been successfully proven in multiple large-scale applications [17, 18]. As no DWC unit is available off-the-shelf in Aspen Plus, it was simulated as a thermodynamical equivalent sequence of distillation columns (see Figure 2). The bottom liquid from the left part of the DWC (DWC_L) is sent to the right part (DWC_R). Simultaneously, part of vapor rising in DWC_R is redirected to DWC_L to ensure sufficient vapor flow in this part of the column. The DWC has 20 stages in total, with the first and the last stages being condenser and reboiler by the convention of Aspen Plus. The wall is placed in the top 14 stages (13 excluding the condenser). Due to the large temperature difference between the BDO and light impurities (top products), thermal insulation will be needed to ensure the energy efficiency of the DWC Reduced pressure operation (top pressure of 0.1 bar) was implemented to avoid high temperatures that may lead to decomposition. Due to the vacuum operation, structured packing was assumed for the DWC's internals. Finally, high purity (>99.4 wt% in all cases) BDO product was recovered as the top product from DWC_R. The remaining light and heavy impurities are separated as the top product from DWC_L and the bottom product from DWC_R. As a worst-case scenario, and due to the low concentrations, it was assumed that water and light impurities would be sent to wastewater treatment, while heavy impurities would be burnt for energy. Appropriate economic and environmental metrics are included in the evaluation of the developed process.

Table 1. Key performance indicators

	Case 2-3	Case 1-4	Case 1-3
Economic indicators			
CAPEX (k$)	9,402	8,419	9,111
OPEX ($/$kg_{BDO}$)	0.159	0.132	0.164
Total annual costs ($/$kg_{BDO}$), 10 years payback period	0.222	0.208	0.243
Sustainability metrics			
Thermal energy requirements ($kW_{th}h/kg_{BDO}$)	0.506	0.662	0.615
Electrical energy requirements ($kW_{e}h/kg_{BDO}$)	0.539	0.502	0.624
Primary energy requirements ($kW_{th}h/kg_{BDO}$)	1.854	1.917	2.176
CO_2 emissions, grey / green electricity (kg_{CO2}/kg_{BDO})	0.319 / 0.073	0.324 / 0.096	0.373 / 0.089
Water consumption (m^3_{w}/kg_{BDO})	0.145	0.145	0.161
Wastewater intensity (m^3_{ww}/kg_{BDO})	0.010	0.014	0.013
Material intensity (kg_{waste}/kg_{BDO})	0.037	0.004	0.024

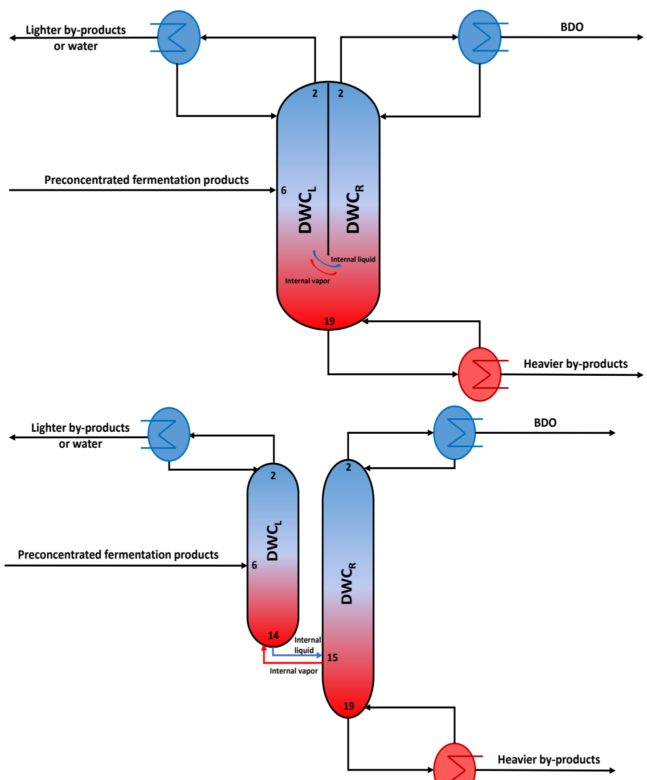

Figure 2. DWC (top) and equivalent sequence of distillation columns (bottom)

Analysis of economic and environmental impact

The performance of the developed recovery processes was evaluated by analyzing economic indicators and sustainability metrics following the published recommendations [19, 20]. A comparison between the three processes is presented in Table 1.

Slightly lower capital costs in case 1-4 are due to marginally lower costs of distillation columns and compressor in the MVR system. Similarly, lower operating costs (OPEX) in case 1–4 are mainly because of lower wastewater treatment and electricity expenses (due to lower amount of light impurities and lower required compressor power in the MVR system). Total annual costs that include both CAPEX and OPEX with a payback period of 10 years are 0.222, 0.208 and 0.243 $/kgBDO in cases 2-3, 1-4 and 1-3, respectively. Thus, despite a somewhat lower initial concentration of 1,4-BDO (6.97 wt%) compared to 2,3-BDO (9.30 wt%) and 1,3-BDO (7.25 wt%), process performance is slightly better due to lower amounts of light impurities. Nonetheless, given that the minimum selling price for 2,3-BDO produced from sucrose, molasses or glycerol has been estimated to be 3.7 – 5.7 $/$kg_{BDO}$ [5], the proposed cost-effective recovery process may present a major advancement toward competitive fermentative production of BDO. Furthermore, there is significant margin compared to the price of petrochemically produced BDOs (about 1.8 $/kg [21]). However, fermentation costs still need to be accounted for to obtain a real picture.

Furthermore, thermal energy requirements are the lowest in case 2-3 (0.506 $kW_{th}h/kg_{BDO}$) due to the largest product flowrate. On the contrary, electrical energy requirements are the lowest in case 1-4 because of the lowest compressor power in the MVR system. The total primary energy requirements were calculated considering a conservative electrical-to-thermal conversion factor of 2.5. This metric ranges from 1.85 $kW_{th}h/kg_{BDO}$ for case 2-3 to 2.18 $kW_{th}h/kg_{BDO}$ for case 1-3, which is much lower than the 3.1 – 6.5 $kW_{th}h/kg_{BDO}$, reported in the literature. Related to energy use are CO_2 emissions, which are again the lowest in case 2-3. Water consumption (including 7 % loss of cooling water and 70 % recovery of condensate in the steam cycle) is also the lowest in case 2-3 due to the lowest thermal energy requirements. Material intensity is the lowest in case 1-4 because of the lowest amounts of light impurities.

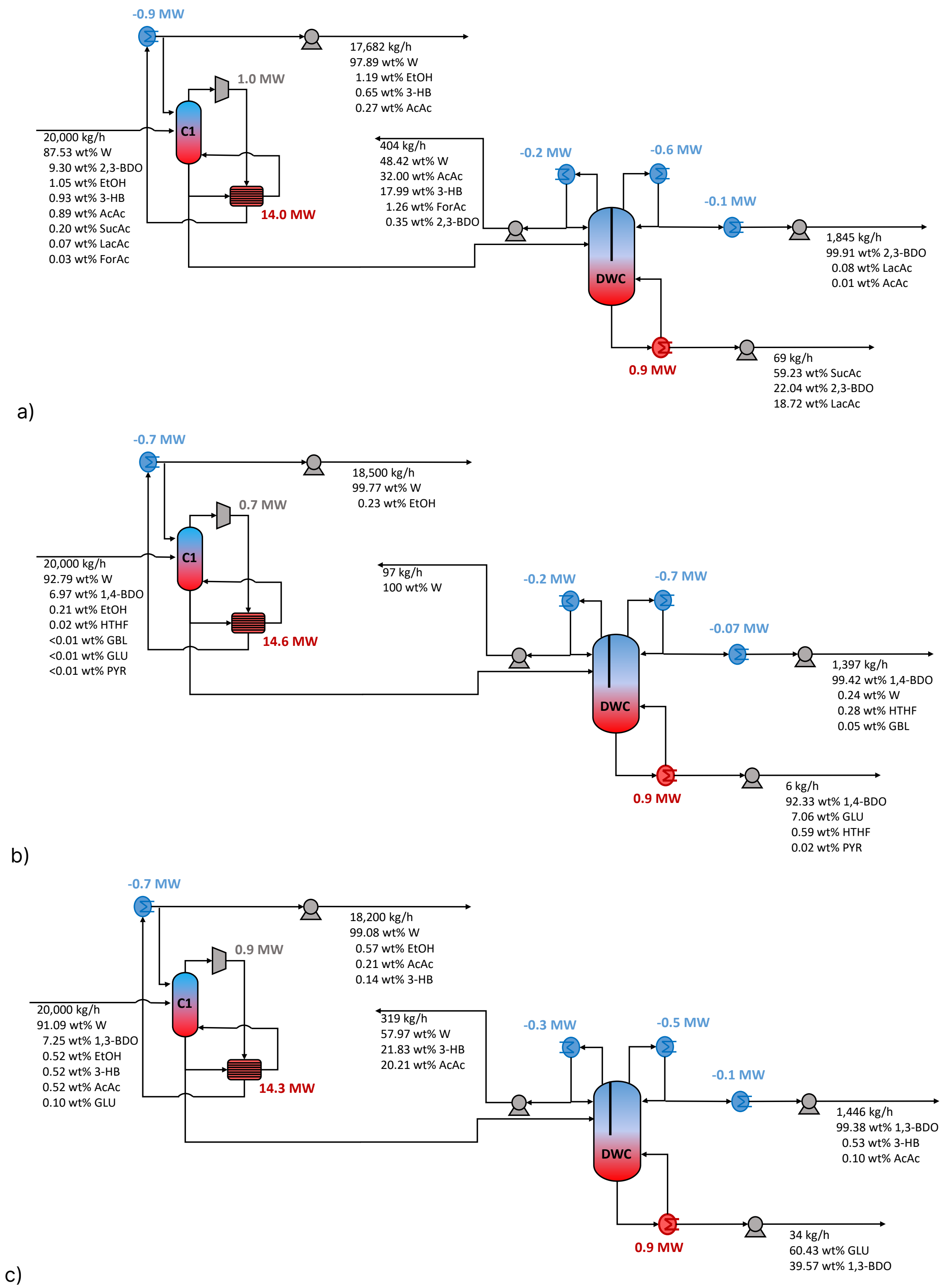

Figure 3. Flowsheet of BDOs' recovery process: a) 2,3-BDO, b) 1,4-BDO and c) 1,3-BDO

CONCLUSION

This original paper proposes an eco-efficient large-scale downstream processing design for the preconcentration and final purification steps in the purification of different BDOs (2,3-, 1,4- and 1,4-) after fermentation. Due to similar fermentation broth compositions and thermodynamic properties, a single process design was proven to cost-effectively (0.208 – 0.243 $/kg_{BDO}$) and energy-efficiently (1.85 – 2.18 $kW_{th}h/kg_{BDO}$) recover over 99% of BDO from different fermentation processes. Implementation of the advanced process intensification and heat integration techniques reduced energy requirements by over 33% compared to the existing literature. Furthermore, the adaptable purification process offers flexibility in developing sustainable business models. Lastly, the results of this novel work highlight the importance of using CAPE tools in developing competitive bioprocesses by demonstrating that computer-aided simulations may play a crucial role in advancing sustainable industrial fermentation.

OUTLOOK

While this study provides valuable insights into the techno-economic and environmental feasibility of the proposed processes for the recovery of BDOs after fermentation, there is room for further research to enhance its applicability and robustness. Although the developed process simulations rely on well-established thermodynamic models and rigorously validated unit operation models in Aspen Plus, experimental validation remains essential to ensure successful industrial implementation. Computational simulations should serve as a guiding framework for experiments, ensuring a more efficient and reliable approach to process development. Additionally, while the feasibility of DWC has been demonstrated in real-world applications, further research is needed to optimize equipment design and control strategies for maximizing operational efficiency.

Expanding the process design to include the fermentation reaction would provide a more comprehensive evaluation of economic competitiveness and enable a fair comparison with existing BDO production methods. Moreover, conducting a sensitivity analysis to assess the impact of variations in key factors—such as feedstock costs, utility prices, wastewater treatment costs, and BDO market prices—would offer deeper economic insights. A full life cycle assessment (LCA) would also be valuable in obtaining a more comprehensive understanding of the sustainability of the entire process.

Furthermore, an interesting avenue for future research is the development of a flexible process capable of producing multiple BDO isomers based on market demand. Given the fluctuating market dynamics of bio-based chemicals, designing a process that can adapt to varying production needs could enhance economic viability and commercial competitiveness.

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