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Feasibility study of polyetherimide membrane for enrichment of carbon dioxide from synthetic biohydrogen mixture and subsequent utilization scenario using microalgae

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Summary

In this work, the potential of utilizing a polyetherimide (PEI) hollow-fiber membrane to separate synthetic biohydrogen mixture (H_2/CO_2) was studied. From the gas separation experiments, where the effects of feed to permeate pressure ratio ($P_{feed}/P_{permeate}$) and stage-cut as key factors were evaluated, it was found that the PEI membrane had the capacity to purify either H_2 or CO_2. It turned out that different separation settings should be chosen in accordance with the actual technological purpose, defined either as the enrichment of H_2 or CO_2. The highest H_2 concentration (66.4 vol%) in the permeate was achieved at $P_{feed}/P_{permeate}$ of 4.62 and stage-cut of 0.47, while the peak CO_2 concentration (79.2 vol%) in the retentate was obtained by applying $P_{feed}/P_{permeate}$ of 4.55 and stage-cut of 0.65. The assessment and discussion of results indicated the possible utilization of the CO_2-rich fraction (produced by the PEI membrane) for the biological sequestration using microalgae. To our knowledge, PEI membranes have not yet been tested in such a concept and thus, the results and experiences can mean a new contribution to the literature.

KEYWORDS

biohydrogen, CO_2 utilization, membrane separation, microalgae, polyetherimide, process integration

1 | INTRODUCTION

Biohydrogen (the H_2 gas generated on microbiological grounds) is an emerging energy carrier and with time, expected to support the decarbonization of energy sectors and contribute to sustainability.[1-4] Although dark fermentative hydrogen production has been shown as a feasible pathway, the actual success of the technology is largely influenced by the organic feedstock properties.[5-7] In the early stages of development, easily digestible and relatively simple, carbohydrate-based substrates such as glucose and starch were mainly deployed to conduct fundamental studies in dark fermentative hydrogen-producing systems.[8] Nowadays, with the intense progression of the field, it is rather trending to shift toward the valorization of complex raw materials especially at practical-scale applications. These materials, on a wider spectrum among others, include agro-industrial-, cafeteria/food- and municipal-wastes, wastewaters.[9-14] Additionally, there have been research wave and strong promotion on the cultivation of microalgae as renewable, so-called third generation biomass sources and their subsequent use of for the biofuel production, including biohydrogen.[15,16] Besides the potential of microalgae to serve as the feedstock for hydrogen fermentation, it was also demonstrated that versatile effluents of dark fermentation bioreactors can be utilized by certain microalgae during their own growth and reproduction cycles,[17] viewed as an option in the wastewater-to-bioenergy and hydrogen biorefinery platforms.[18,19] This gives a possibility for the construction of circular bioprocess in which H_2 is evolved and microalgae are grown, according to the scheme demonstrated in Figure 1.[20] Another point to be considered for such an internal, closed-loop design presented in Figure 1 is the purification of biohydrogen gas and the simultaneous valorization of the gaseous by-product with increased CO_2 content. These steps can be assisted by gas separation membranes that bridge the biohydrogen fermenter and the algal photobioreactor and as an advantage, enable for the biological sequestration and

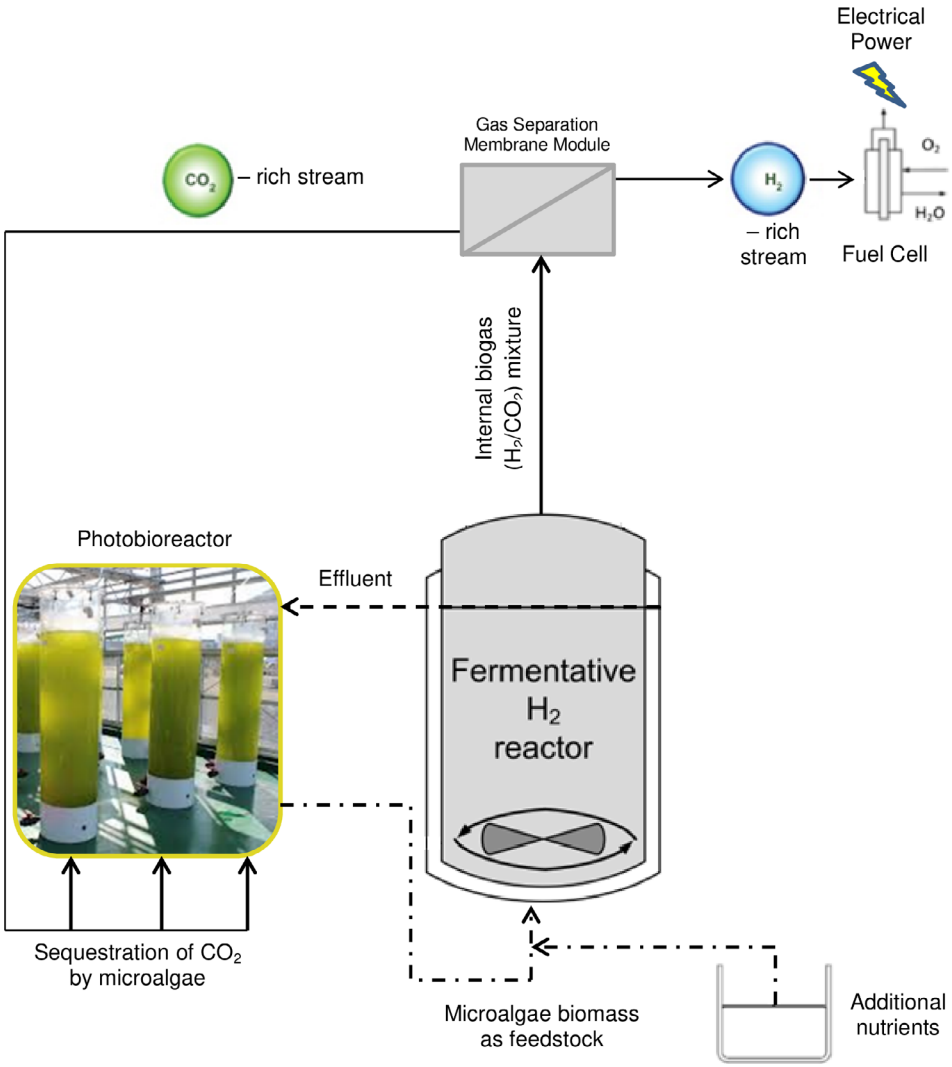

FIGURE 1 Scheme of a circular, internal-loop process for integrated biohydrogen production, separation and valorization of CO_2 to cultivate microalgal biomass as fermentation feedstock [Colour figure can be viewed at wileyonlinelibrary.com]

conversion of carbon dioxide into biomass and other value-added components within the integrated system.[21] However, even if membranes and their modules are carefully selected and operated for the purification of H_2 from the CO_2-rich gaseous mixture formed in the fermenter, achieving full separation is a hurdle and rather, an unlikely perspective. Consequently, in most cases under any permeation conditions, the H_2-concentrated product gas will still contain CO_2 impurities and likewise, the CO_2-enriched side-product stream will carry a fraction of H_2.[22] Eventually, the behavior of the gas separation membrane process can be characterized based on the compositions of the retentate and permeate flows and their relations to the feed gas quality, which provide an important and rapid feedback pertain to the capabilities of a particular membrane for H_2/CO_2 separation and enlighten the favorable operating strategy.

In this study, we describe and assess the experiences of H_2/CO_2 separation by a polymeric, nonporous gas separation membrane fabricated from polyetherimide (PEI) and cast into hollow-fibers, which is a favorable arrangement to get a compact membrane with enhanced gas permeation surface area. The PEI, as stated in the literature, is one of the most promising materials to be used in membrane gas separation thanks in general to its beneficial selectivity features, high thermal resistance, reasonable cost and excellent membrane forming properties.[23] The experiments were conducted with synthetic biohydrogen mixture comprised of hydrogen and carbon dioxide gases considered representative of the H_2 and CO_2 contents generally found in relevant, dark fermentative anaerobic bioreactors. The effects of key parameters, such as feed pressure and stage-cut were investigated on the PEI membrane module and the compositions of gaseous streams leaving the membrane module were monitored. In the end, we discussed the results and concluded the potential of PEI membrane module for a real hydrogen fermentation process, which will be the ultimate goal.

The novelty of this research arises from the testing of PEI membrane by an experimental approach that yield new data for membraneologists and biotechnologists dealing with the design of hybrid concepts for concomitant biohydrogen production, separation as well as cultivation of microalgae on fermentation side products, such as through the valorization of CO_2 originated from the process itself.

2 | MATERIALS AND METHODS

2.1 | Membrane material

PEI was obtained from Sabic under commercial name ULTEM 1000. The formula of a repeated unit of ULTEM 1000 is shown in Figure 2A. Granules were dried at 105°C for 24 hours before use. Solvent for the PEI was *N*-methyl-2-pyrrolidone (NMP) with purity 99.5% delivered by Sigma-Aldrich. Nonsolvent component of spinning solution was absolute ethanol supplied by Penta Chemicals, Czech Republic.

2.2 | Fabrication and characteristics of the PEI membrane module

Hollow-fibers and membrane module was prepared utilizing the infrastructure of the MemBrain company in Czech Republic. Hollow fibers were prepared by phase inversion process method.[24,25] A solution containing 27 wt% PEI, 12 wt% EtOH and 61% by weight of NMP was used to make hollow-fiber membranes. This solution was spun using a spinning line at 50°C into a precipitation bath filled with RO water with temperature of 20°C. During the production of membranes the polymer solution flow was 2.7 mL/min, bore liquid flow was 3.0 mL/min, distance of spinning nozzle from the water surface (air gap) was 9 cm and the towing speed of precipitated fiber was 17 m/min. Fiber was then towed into the continuous washing bath an finally collected on winding wheel. Subsequently, the residual solvent was washed out from the hollow fibers in tanks using large amount of water (24 hours), ethanol (24 hours) and hexane (24 hours). After thorough drying, the surface of hollow fibers was coated with thin layer of silicone rubber (using 1 wt% solution of PDMS in hexane, crosslinked and dried at 70°C for 12 hours) to protect the separation layer on the outer surface of the fiber and to cover microscopic defects in the fiber. The resulting hollow-fiber membrane (Figures 2A and 3) had regular rounded shape with the sponge-like structure containing typical finger-like pores of relatively small size (<20 μm). As determined using the optical microscope, the outer and inner diameters of the hollow fibers were 349 ± 9 μm and 172 ± 5 μm, respectively.

Hollow fibers were arranged into a bundle of 400 fibers (membrane area: 1200 cm^2) and introduced into a PVC pipe and at the ends of the pipe the fibers were sealed using specially designed epoxy resin. After 48 hours, when the resin was completely crosslinked, the end of the pipe was cut with the special blade to make hollow part of the fibers accessible. Subsequently, the PVC flanges were welded from the outer side at the ends of the pipe. To these flanges, the stainless steel flange counterpart was attached over the Viton o-ring. Finally, the 6 mm Swagelok connectors were attached to the flanges for easy connectivity to testing unit.

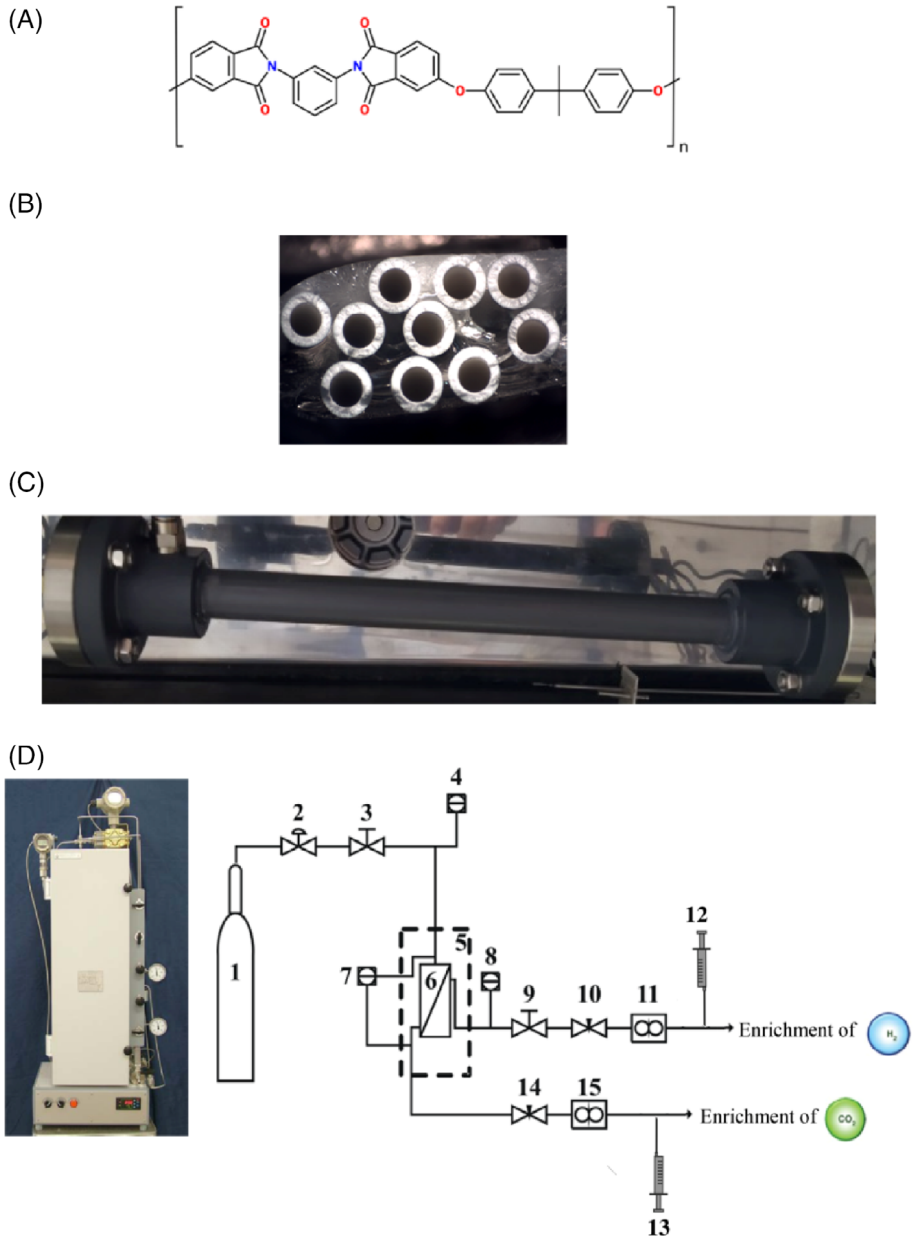

FIGURE 2 The material, A, hollow fibers, B, module, C, of the polyetherimide (PEI) membrane and, D, the experimental membrane testing GSMS-100 apparatus. Notation numbers in, D, are explained in Section 2.2 [Colour figure can be viewed at wileyonlinelibrary.com]

2.3 | Gas separation setup

In this work, the evaluation of the membrane module containing PEI hollow fibers (Figure 2A-C) was performed in the GSMS-100 gas separation test rig described by Bakonyi et al[26] and Nemestóthy et al[27] with scheme presented in Figure 2D. In brief, the GSMS-100 system is able to control (by regulator "2" and valve "3") the pressure and flow of the gas fed (from gas container "1") to the membrane ("6"). The actual feed pressure is indicated by gauge "4." The membrane is kept in the temperature regulated chamber "5" (28°C in this research). The actual difference between the feed and retentate pressures can be measured by gauge "7." In the permeate line, the pressure (atmospheric in this study) is displayed by gauge "8," the flow is controlled by valves "9" and "10" and measured by mass flow meter "11." In the retentate line, the flow can be adjusted and monitored by valve "14" and mass flow meter "15," respectively. Basically, the whole GSMS-100 device contained a stainless steel pipeline where analog pressure gauge "8," valves, connectors and fittings were purchased from Swagelok. The digital pressure gauges "4" and "7" were bought from Honeywell. The mass flow meters were products of the company Bronkhorst High-Tech B.V. (The Netherlands) and were subjected to preliminary calibration.

Following the installation of PEI membrane module into GSMS-100, the gas separation measurements were commenced after successful leakage tests and the adequate integrity of the setup is reflected by the fair (<5%)

mass balance errors, as shown in Table 1 together with the experimental plan.

In Table 1, it can be seen that the effects of (a) the feed to permeate pressure and (b) the stage-cut (ratio of the permeate flow to the total flow) were investigated. The binary feed gas containing (45.2 vol%) H_2 and (54.8 vol%) CO_2 was prepared in a gas cylinder and employed as a synthetic H_2 fermentation mixture. Accordingly, this setup was designed to conduct experiment off-line, separately from a bioreactor ("hypothesized integrated system"), which can be a next step based on the preliminary feedback delivered from this study. The PEI membrane module was operated in lumen-to-shell separation arrangement, where the gas is fed into the hollow fibers and retentate is obtained on the opposite end of the capillaries, while permeate, after passing through, is collected on the shell side. The actual composition of gas samples taken under steady-state permeation conditions via ports "12" and "13" was determined using gas chromatography method reported elsewhere.[28]

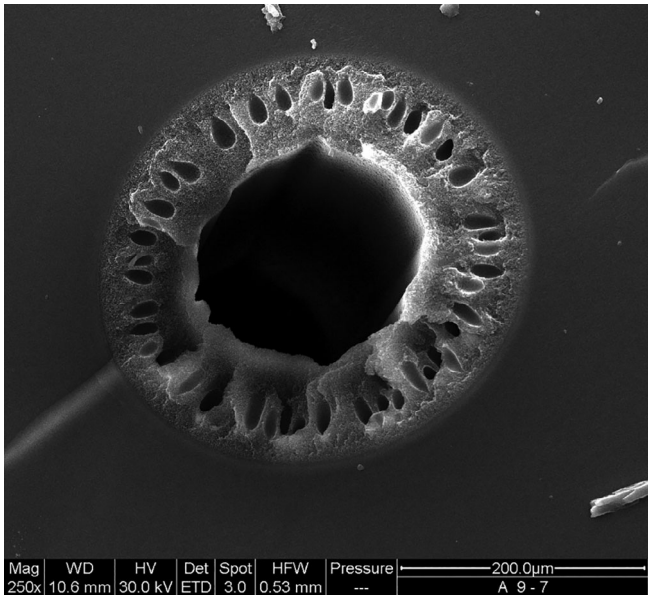

FIGURE 3 Microstructure of the polyetherimide (PEI) hollow-fiber membrane cross-section obtained from scanning electron microscopy (SEM)

3 | RESULTS AND DISCUSSION

As foreshadowed in Figure 1, the final goal with the application of PEI membrane will be the utilization of CO_2-enriched gas fraction (derived from the fermentation) for the cultivation of microalgae. At the same time, a H_2-rich fraction should continue its path toward fuel cells. Thus, the objective of the current process evaluation is twofold and should be concerned both with $H_2$and CO_2 purifications.

The numerical results obtained with the PEI membrane in the respective experiments are shown in Table 1. As it can be concluded from the observed behavior of the membrane made of glassy polymer PEI,[23] this material is H_2-selective (or in other words, CO_2-rejective) in coincidence with earlier investigations.[29,30] According to the gas composition data in Table 1, higher concentrations of H_2 were achievable in the permeate (shell side of the module). Furthermore, from the response surface in Figure 4A, which is fitted to the experimental results of Table 1 to illustrate trends, it can be drawn that the purity of H_2 in the permeate was notably affected by both the (a) feed to permeate pressure ratio ($P_{feed}/P_{permeate}$) and (b) the stage-cut, as separation settings. Similar observations can be made regarding the role of these two separation variables on the enrichment of CO_2 on the opposite, (lumen) side of the module in the retentate (Figure 4B).

Although in both cases (Figure 4A,B) the positive effect of higher $P_{feed}/P_{permeate}$ could be noticed that is in link with the driving force of the separation, the impact of stage-cut is somewhat more complex. As a matter of fact, lower stage-cuts (lower quantity permeate, more retentate) are favored for purifying H_2 in the permeate

TABLE 1 The experimental plan for testing the PEI membrane module with binary H_2/CO_2 mixture and the gas composition results obtained accordingly

$P_{feed}/P_{permeate}$ (1)	Stage-cut (1)	Gas compositions (vol%)						Mass balance error (%)	
		Feed		Retentate		Permeate			
		H_2	CO_2	H_2	CO_2	H_2	CO_2	H_2	CO_2
2.45	0.13	45.2	54.8	43.1	56.9	65.8	34.2	1.9	1.6
2.45	0.41	45.2	54.8	35.5	64.5	64.0	36.0	4.3	3.6
2.55	0.57	45.2	54.8	28.0	72.0	61.0	39.0	3.8	3.1
4.55	0.65	45.2	54.8	20.8	79.2	61.1	38.9	4.2	3.4
4.62	0.47	45.2	54.8	29.4	70.6	66.4	33.6	3.4	2.8

Note: $P_{permeate}$: 1 atm (absolute).
Abbreviation: PEI, polyetherimide.

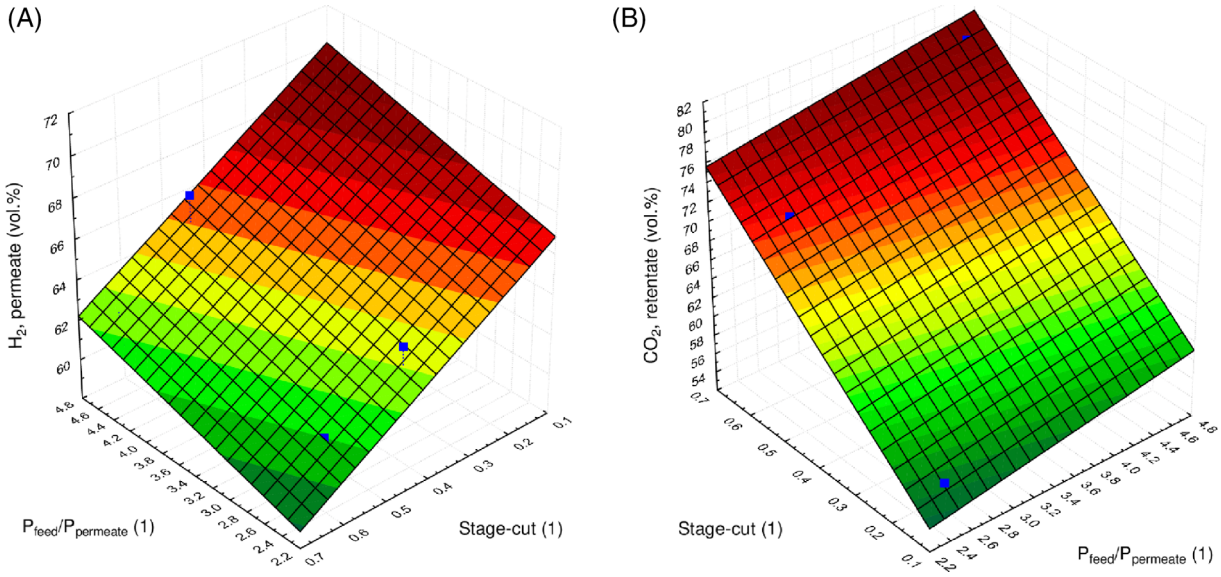

FIGURE 4 The effect of gas separation conditions on the variation of, A, H_2 concentration in the permeate and, B, CO_2 concentration in the retentate applying the polyetherimide (PEI) membrane module [Colour figure can be viewed at wileyonlinelibrary.com]

(Figure 4A), while higher stage-cuts (higher quantity permeate, less retentate) seem required to enhance the CO_2 concentration in the retentate (Figure 4B). This is well-reflected by examining the first two lines in Table 1 at equal $P_{feed}/P_{permeate}$ ratios (2.45), where decreasing the stage-cut from 0.41 to 0.13 resulted in the increment of H_2 concentration in the permeate (from 64 to 65.8 vol%), while reduced the CO_2 content in the retentate (from 64.5 to 56.9 vol%).Therefore, in agreement with the common experiences and supported by our recent calculations carried out on PEBAX gas separation membrane,[20] simultaneously targeting the purifications of H_2 and $CO_2$is not a feasible option and a decision is needed which way to go with the adjustment of permeate and retentate flows to meet the actual preferences. In this study employing the PEI membrane and a constant feed gas quality mimicking a realistic mixture of biohydrogen fermenters (Table 1); on the one hand, the highest H_2 concentration of 66.4 vol% in the permeate was attained at $P_{feed}/P_{permeate}$ and stage-cut of 4.62 and 0.47, respectively. If the enrichment of H_2 was aimed, then the product gas with increased H_2 content should pass toward application in fuel cells to produce electricity, such as it was demonstrated by Lin et al.[31] On the other hand, the suitable conditions for the peak CO_2 concentration (79.2 vol%) in the retentate were $P_{feed}/P_{permeate}$ and stage-cut of 4.55 and 0.65, respectively. In this latter case, as an example, the mixed gas (H_2/CO_2) selectivity of the membrane, calculated as the ratio of component permeances (H_2:172.2 and CO_2:36.8, both in the unit of 10^{-6} cm^3_{STP} cm^{-2} s^{-1} cm Hg^{-1} known as GPU),[32,33] was found to be 4.68. For comparison, we may recall the findings of previous studies investigating the performance of PEI material for H_2/CO_2 separation. Actually, H_2/CO_2 selectivity values spanning 5.9 to 6.4 were reported applying feed pressures up to approximately 10 atm and a temperature of 30°C to 35°C.[29,30,34] It should be, however, noted that these results originate mostly from single gas measurements that are considered ideal and hence, are usually better than those obtained with a gas mixture. It can be extracted from the literature that the concentration of CO_2 in the gas entering the photobioreactor can have notable impact, depending on the tolerance of various species to elevated CO_2 concentrations. As analyzed by Singh and Singh for several microalgae, for example, *Chlorella*, *Scenedesmus*, *Nannochloropsis*,[35] frequent concentration levels of CO_2 in the gas supplied for biomass production are up to 15%. Nevertheless, certain species, can be cultivated with compromises even under CO_2 concentrations of 50%,[36] 70%[37] or even 80%, while 100% likely causes complete inhibition.[38] Overall, in line with these previous findings, it can be argued based on the assessment of PEI membrane that the module has the potential to contribute to the management of H_2/CO_2 separation during biohydrogen fermentation and ensure a CO_2-rich, secondary gas stream (up to 79.2 vol% under the conditions tested in this work) that may be transformed biologically in an optimized photobioreactor system, provided that the underlying microalgae species are carefully selected. In the future, based on all of these considerations, it is proposed to implement the integrated system using a real hydrogen gas fermenter and an algal photobioreactor and explore its operation, readiness level and energy balance at a larger, postlaboratory scale.

4 | CONCLUSIONS

In this study, a PEI membrane module was evaluated for its H_2/CO_2 separation capability. It was demonstrated in the experiments that the gas separation task could be managed by the membrane, however, different operating settings needed to be adjusted for purifications of H_2 and CO_2 gases. Under appropriate conditions, a CO_2-enriched retentate with 79.2 vol% of CO_2 was delivered and its photobiological transformation pathway employing microalgae cultures was discussed considering an integrated system, where carbon dioxide is captured and converted into biomass as the renewable feedstock of an envisaged circular bioprocess producing biohydrogen fermentatively. We also see significant improvement potential in the optimization of the membrane and the fabrication of the module or in special membrane materials tailored for the separation of H_2/CO_2 from biological sources.

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