

Research Article

The Effect of Sweeping Media and Temperature on Aqueous CO₂ Removal Using Hollow Fiber Membrane Contactor (HFMC): An Experimental Determination

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Transport phenomena through hollow fiber membrane contactors (HFMCs) indicate the exchange of a component between the two phases, inside and outside of hollow fibers. In this research, we designed and fabricated lab-made HFMCs to assess the difference between water and air as sweeping media for CO₂ exchange. The effects of flow rates and temperature ratios on aqueous CO₂ absorption were investigated accordingly. A semiclosed circuit incorporating our fabricated HFMCs was set up to regulate the operating parameters and evaluate the aqueous CO₂ concentration using an initiative pH-based method. The results of our experiments remarkably reveal that air tends to remove aqueous CO₂ more than water when aqueous CO₂ concentration is higher than 3.53×10^{-6} mlCO₂/l. However, water would surpass air in lower concentrations. Nevertheless, tripling the flow rate of sweeping media from 500 to 1500 ml/min shifts up this cutoff point 50 times to around 1.66×10^{-4} mlCO₂/l. The experiments performed at three different temperature ratios of 22 : 22, 44 : 12, and 22 : 12°C (CO₂-rich liquid: sweeping medium) demonstrated that a higher temperature gradient deteriorates the CO₂ absorption capacity of sweeping media. Nonetheless, temperature gradient becomes highly effective in aqueous CO₂ concentrations lower than 1.57×10^{-6} CO₂/l. The results of this research could be applied in performance optimization of aqueous CO₂ absorbing HFMCs, even in sophisticated medical procedures such as arterio-venous and veno-venous CO₂ removal systems where both water and air could be used as blood's CO₂ sweeping media.

1. Introduction

Nowadays, membrane contactors are widely used in several chemical or biological processes, including separations and reactions [1–5]. Among them, hollow fiber membrane contactors (HFMCs) are commonly employed in medical, pharmaceutical, biological, and environmental applications. Extracorporeal membrane oxygenation (e.g., biohybrid oxygenators [6–8], extracorporeal CO₂ removal (ECCO2R) [9, 10], arterio-venous CO₂ removal (AVCO2R) and veno-venous CO₂ removal (VVCO2R) devices [11–13]), hemodialysis [14, 15], tissue culturing [16], membrane bioreactors used for wastewater treatment [17–20], air purification (e.g., flue gas absorbing and CO₂ capturing [21–27]), and reverse osmosis applied in, e.g., water purification [28] and

concentration process [29] are various examples where HFMCs are employed as one of their major components. In several applications, such as AVCO2R and VVCO2R, aqueous CO₂ removal is considered the significant performance objective of HFMCs [30].

HFMCs can be operated as liquid-liquid [31, 32], gas-liquid [33], or gas-gas [34], depending on the fluids flowing inside and outside of hollow fiber lumens.

Various parameters affect the efficiency of the CO₂ removal process, such as module configuration, hollow fiber arrangement, type of sweeping medium, temperature, and fluid dynamics such as the flow rate, the temperature of fluids, the flow pattern, and the CO₂ concentration in both media (i.e., absorbing/sweeper and removing/carrier media) [35–37]. However, no specific publication addresses the

effects of the parameters mentioned previously on aqueous CO₂ removal.

In this article, the affinity of water to capture aqueous CO₂ was investigated and compared with air through identical setup designs. In addition, the effect of fluid dynamics and the temperature of the sweeper and carrier media were experimentally examined. This research uses the HFMC as an aqueous CO₂ removal device. The CO₂-containing liquid flows outside the hollow fiber bundle, and the sweeping media of air or water absorbs CO₂ by flowing inside the lumens. The results of this research bring light on the difference between water and air as a sweeping medium with respect to their ability to remove aqueous CO₂. This could eventually be used in design modifications of HFMCs in AVCO2R and VVCO2R devices.

2. Materials and Methods

2.1. Equipment and Materials. In order to investigate the effect of sweeping media and temperature on the removal of the aqueous CO₂, different experimental circuits had been designed and set up. The experiments were based on the principle of CO₂ dissociation in water and the resulting aqueous pH drop. The following equipment and materials were utilized in experimental setups to measure the pH change as a consequence of CO₂ exchange in testing HFMCs: Stöckert SIII including 2 roller pumps (Stöckert Instrumente GmbH, Germany), PT-100 temperature sensors connected to the Stöckert temperature measuring system (Stöckert Instrumente GmbH, Germany), liquid flow meter T110 ultrasonic sensor (Transonic System INC., USA), CO₂ and compressed air (Linde AG, Germany), gas flow meters (Brooks Instrument Inc., USA), PVC and silicon tubes and connectors (Raumedic AG, Germany), Capiox® bubble trap (Terumo Cardiovascular Group, USA), and magnet stirrer C-Mag HS7 (IKA-Werke GmbH & CO. KG, Germany). To set the intended temperatures, Omnitherm™ heat exchangers (SciMED Life Systems Inc., USA) were employed in connection with the Stöckert cooling system (Stöckert Instrumente GmbH, Germany) for temperature reduction and HAAK water bath (Thermo Fisher Scientific, USA) for temperature increase with regard to the ambient condition. Distilled and deionized ultrapure water were prepared by employing Milli-Q Advantage A10 ultrapure water purification system (EMD Millipore Corp, Billerica, USA). For pH measurement, FPH-BTA Tris Compatible Flat pH sensors in combination with the compatible data acquisition board LabQuest Vernier and Logger Lite® v.1.6.1 software (Vernier Inc., USA) were used to acquire pH data and send it to a pc by a USB port. The acquisition frequency for pH sensor was set to 2 Hz (i.e., 2 data/s).

2.2. Fabrication of HFMCs. A cylindrical hollow-fiber membrane contactor was designed and fabricated in our laboratory, as indicated in our previous publication [38], with some modifications. Mats of Oxyphan® hollow fiber membranes (Membrana AG, Germany) with custom-made length were ordered. These mats were arranged in the

intermediate space of two concentric cylinders made of polycarbonate polymer (refer to Figure 1(a)). The initial designs were performed with Autodesk Inventor® 2012 (Autodesk Inc., USA) software, which can be used by milling machines for prototypes' construction. Then, the designs were imported to SolidWorks® v.2012 X64 (Dassault Systèmes SolidWorks Corp., France) software to investigate the geometric parameters of module designs in more detail. Using SolidWorks®, the illustration of the whole design, cross sections, and fluid fields were also tested. The basic 3D design of this HFMC created with SolidWorks® is depicted in Figures 1(b) and 1(c).

The hollow fibers in prototypes were then potted by polyurethane glue (Henkel AG & Co. KGaA, Germany), using a custom-built centrifuge device (Wichelhaus GmbH & Co. KG Maschinenfabrik, Germany) as shown in Figure 2(a). The fabricated HFMC is demonstrated in Figures 2(b) and 2(c).

The effective membrane surface area of this HFMC is 0.8 m², while the priming volume is 0.7 × 10⁻⁴ m³. This hollow-fiber membrane contactor could be considered as either liquid-liquid or gas-liquid HFMC, provided that water or air are used as the medium flowing inside hollow fiber lumens, respectively.

While fabricating the HFMC, some challenges and obstacles need to be resolved such as winding the Oxyphan® mat homogenously around the inner shaft of the contactor. To overcome this obstacle, specific pinches and handles were designed and constructed to properly mount the fiber mat on the special lab-made winding machine. This machine induces a constant tension force over the fiber mats; therefore, they can be wound uniformly.

Other than that, the viscosity of the polyurethane glue for potting the hollow fibers via centrifugal force plays an important role in the uniform total sealing of the hollow fibers. The optimum viscosity of the glue was achieved by experience. After potting, both ends of the hollow fibers should be cut in a way that the ends of the fibers remain open which can be challenging and needs experience; hence, the hollow fibers were cut using a very sharp stainless-steel blade.

2.3. Method of Experiments

2.3.1. Liquid-Liquid HFMC. As demonstrated in Figure 3, the setup was designed to measure and compare aqueous CO₂ removal at different temperatures using deionized (DI) water as a sweeping medium. As shown in Figure 3, on the one hand, liquid1, which is CO₂-rich DI water, flows outside the hollow fibers of testing HFMC in a closed loop using the roller-pump1. On the other hand, liquid2, which is CO₂-free DI water, streams counter-currently inside the hollow fiber membrane lumens, in an open circuit using the roller-pump2. In this way, liquid2 removes the CO₂ content of liquid1 gradually resulting in the increase of liquid1's pH.

Before initiation of the experiment, in order to provide CO₂-rich DI water, air bubbles in the closed circuit of liquid1 should be withdrawn properly with the aid of the bubble

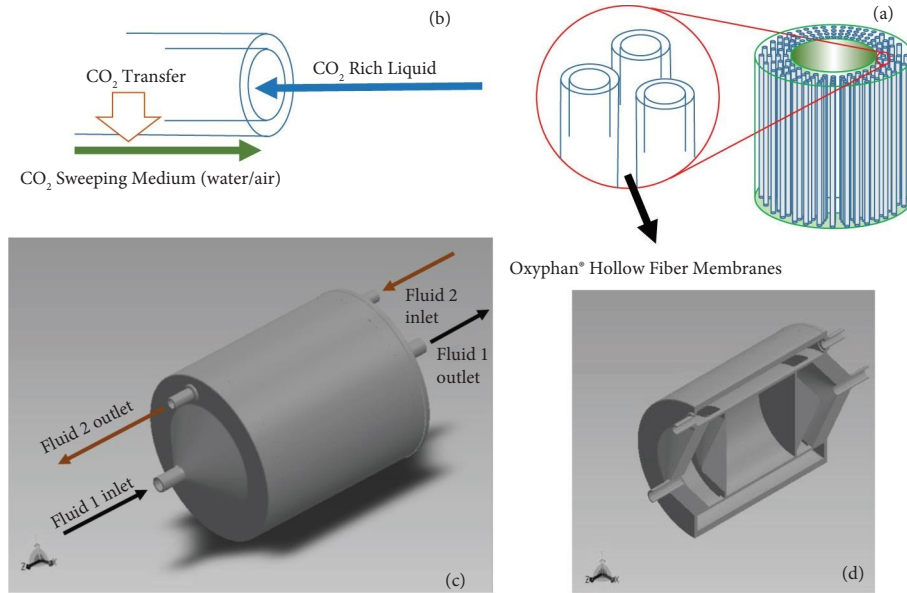


FIGURE 1: Hollow fiber arrangement in the HFMC module, (a) schematic of the CO₂ removal process, (b) 3D top, (c) and cross-section, (d) views of HFMC design and fluid directions.

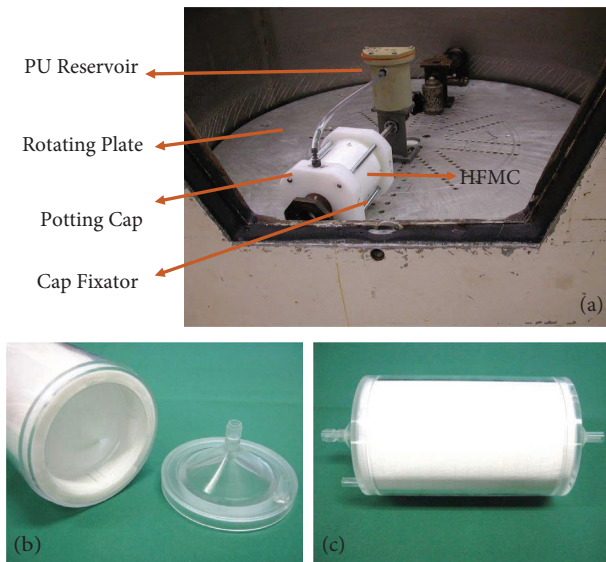


FIGURE 2: Custom-built centrifuge machine potting hollow fibers, (a) cross-section, (b) and top (c) views of our fabricated HFMC.

trap. Then, pure CO₂ was directly inserted into the DI water in reservoir1. Gasification was maintained until the pH in reservoir1 reached a defined value. In addition, a magnet stirrer was used in order to homogenize the CO₂ distribution in the reservoir. The method for the calculation of total aqueous CO₂ concentration based upon aqueous pH was explained in our previous publication [38].

In the first series of liquid-liquid experiments, the flow rate of CO₂-rich DI water was kept constant at 2800 ml/min which is the maximum applicable flow rate of our fabricated HFMCs. The flow rate of the CO₂ sweeping medium, i.e., liquid2, was set at 500, 750, 1000, and 1500 ml/min. Due to the limitation of the liquid flow rate

inside hollow-fiber membranes, the maximum applicable flow rate of liquid2 is 1500 ml/min. The temperatures of both liquids were kept constant at 22°C utilizing both heat exchangers 1 and 2.

The second series of experiments were designed in order to investigate the effect of temperature variation on aqueous CO₂ removal. Here, the flow rates of liquid1 and liquid2 were kept constant at 2800 and 750 ml/min, respectively. The temperatures of liquid1 to that of liquid2 ($T_{liq1} : T_{liq2}$) were set at 44 : 12 and 22 : 12°C. The results were then compared with the corresponding investigation from the first series of experiment where $T_{liq1} : T_{liq2}$ was 22 : 22°C.

2.3.2. Gas-Liquid HFMC. Since Oxyphan® hollow fiber membranes are highly porous, both liquid and gas can pass through and therefore are used in gas-gas, gas-liquid, and liquid-liquid HFMCs. In order to compare the affinities of water and air in removing aqueous CO₂, in the first approach, liquid2 should be replaced by air, as the sweeping medium. As shown in Figure 4, the air flows inside the hollow-fiber lumens as a sweeping medium, while its flow rate is adjusted using the Brooks air flow meter and CO₂-rich liquid flows counter currently outside the lumens in a closed loop using the roller-pump1 as well as the liquid-liquid HFMC. This converts the HFMC into a gas-liquid contactor. In gas-liquid HFMC, the air is used to remove the aqueous CO₂ gradually which leads to the increase in pH. All other conditions are the same as liquid-liquid experiments as mentioned previously. Briefly, in the first series of gas-liquid experiments, the liquid flow rate was kept constant at 2800 ml/min and the air flow rate was set at 500, 750, 1000, and 1500 ml/min. Temperatures of both liquids were kept constant at 22°C utilizing both heat exchangers 1 and 2.

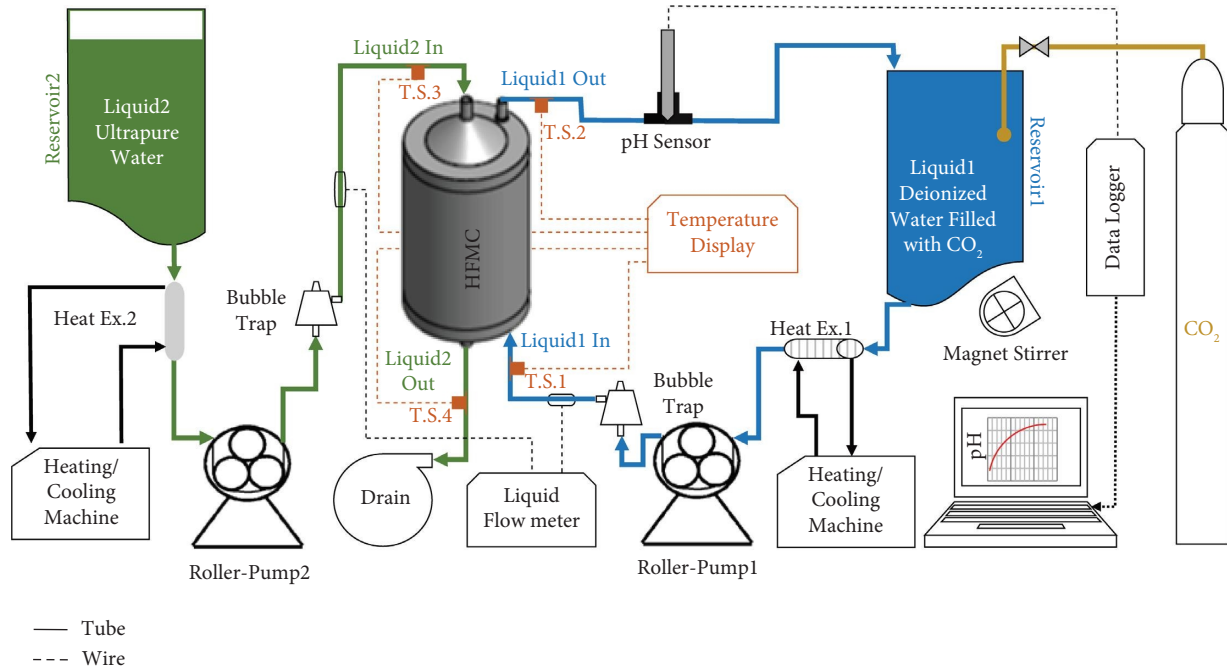


FIGURE 3: Schematic demonstration of a liquid-liquid HFMC experiment with temperature regulations.

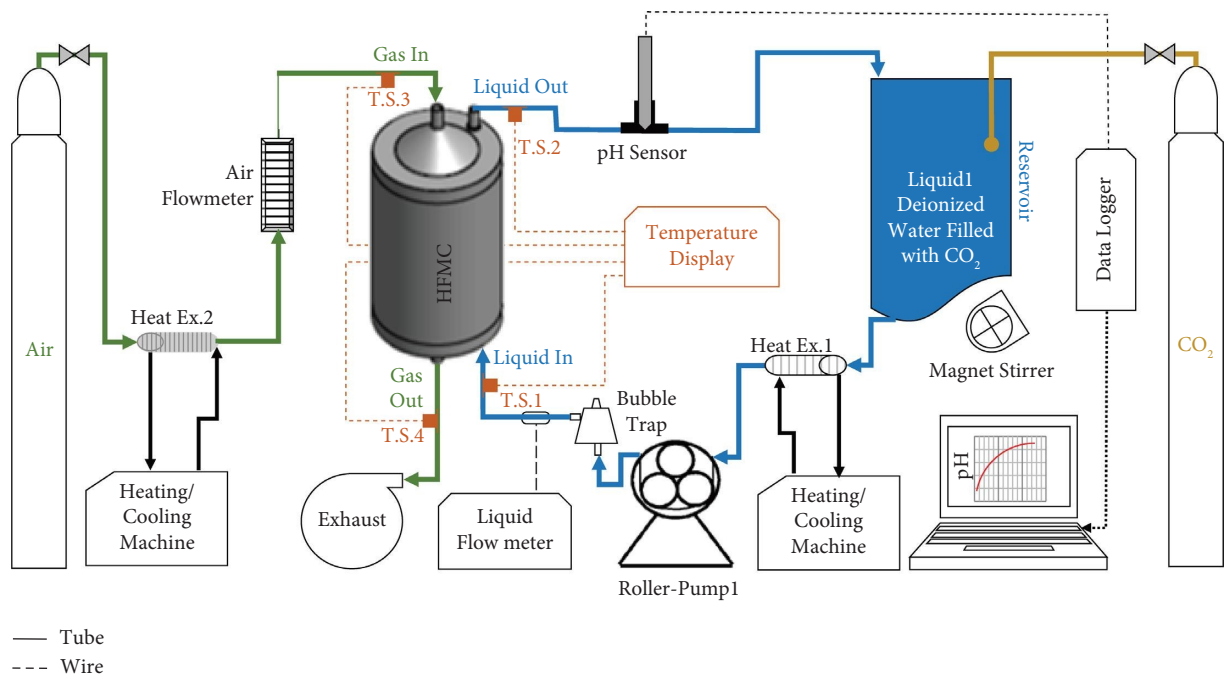


FIGURE 4: Schematic demonstration of air-liquid HFMC experiment with temperature regulations.

In the second series of experiments, the flow rates of liquid and air were kept constant at 2800 and 750 ml/min, respectively. The temperatures of CO_2 -rich liquid to that of air ($T_{\text{liq}}:T_{\text{air}}$) were set at 44:12 and 22:12°C. The results were then compared with the corresponding investigation from the first series of experiments where $T_{\text{liq}}:T_{\text{air}}$ was 22:22°C.

3. Results

3.1. Effect of Sweeping Media on Aqueous CO_2 Removal. In order to investigate the effect of sweeping media on aqueous CO_2 removal, both liquid-liquid (shown in Figure 3) and gas-liquid (shown in Figure 4) circuits were run under the same operating conditions and the relevant

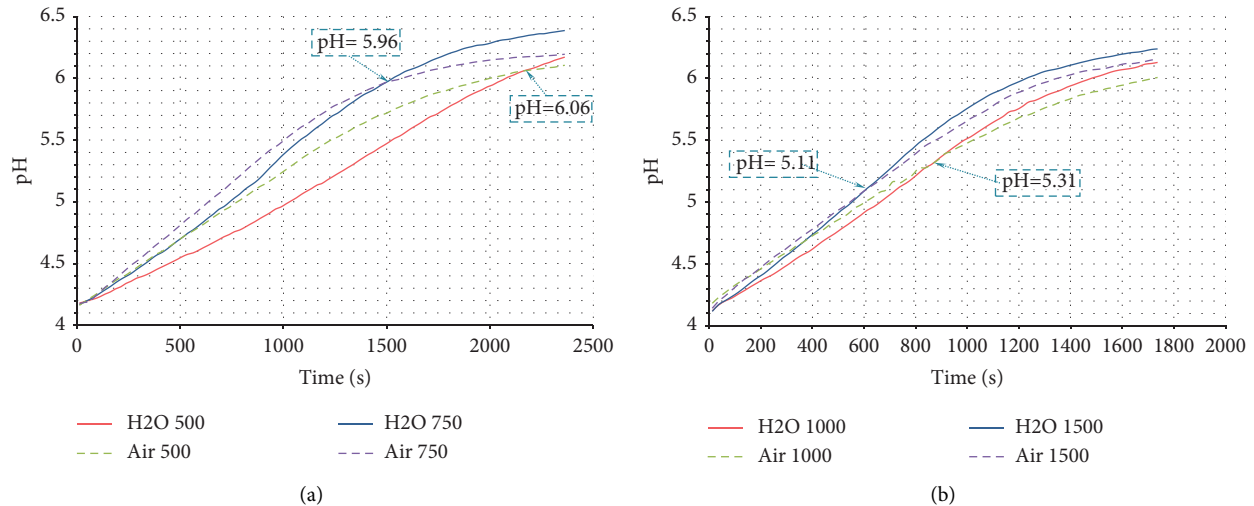


FIGURE 5: pH variations as a result of CO₂ removal by air and water at flow rates of 500 and 750 ml/min (a) and 1000 and 1500 ml/min (b).

pH variations were recorded, while the flow rate of liquid1 was kept constant at 2800 ml/min, and the flow rate of fluid2 (water and air in liquid-liquid and gas-liquid circuits, respectively) varied from 500 to 1500 ml/min. To initiate these experiments, liquid1 was gasified with CO₂, resulting in a pH value of around 4.2. Herein, air or liquid2 would sweep the aqueous CO₂, and consequently, the pH level of liquid1 would increase. Figure 5 depicts the results of pH raise using water and air as the sweeping media with the flow rates of 500, 750, 1000, and 1500 ml/min. The subsequent aqueous CO₂ removal capability could be calculated by the pH-based method [38].

To compensate the fluctuations in the recorded pH values and demonstrating smoothen curves in this work, a moving average function was applied for them. Each series of experiments was conducted 3 times. As shown, the recorded pH courses have the same trends as in Figure 5. The intersection points of the corresponding pH courses using water or air as the CO₂ sweeping media are also shown in Figure 5 (indicated pH values for the intersection points are the mean pH of three experiment repetitions).

3.2. Effect of Temperature on Aqueous CO₂ Removal. In order to investigate the temperature effect on aqueous CO₂ removal, experiments were conducted, while the flow ratio of the two fluids was adjusted at $Q_{liq1} : Q_{liq2/air} = 2800 : 750$ ml/min, and the temperatures of liquid1 to that of liquid2 or air $T_{liq1} : T_{liq2/air}$ were set at 44 : 12 and 22 : 12 °C. The results were then compared with experiments using $T_{liq1} : T_{liq2/air}$ of 22 : 22 °C. The set temperatures ($T_{T.S.1}$ and $T_{T.S.3}$) and the steady state recorded ones ($T_{T.S.2}$ and $T_{T.S.4}$) for these experiments are displayed in Tables 1 and 2. The results are reported as an average of three repetitions with their standard deviations (SD). The outlet temperatures became steady in less than 20 seconds for the adjusted flow rates. The curves of pH change for these experiments on liquid-liquid and gas-liquid HFMCs are depicted in Figure 6.

4. Discussion

The difference between water and air in regard with their ability to remove the aqueous CO₂ by using a liquid-liquid and gas-liquid HFMC has not yet been reported. Although studies show that different pressures' availability of water has different effects on CO₂ removal from CO₂-rich gas. At subatmospheric pressure, increasing humidity leads to less CO₂ uptake, and at high pressure, more CO₂ uptake is achieved by increasing humidity [39].

An expectable outcome according to the literature which indicates that the flow rate of the sweeping medium is in correlation with CO₂ removal [40, 41] was observed. The curves demonstrated in Figure 5 show that the higher the flow rate of fluid2, the higher the rate of pH increase in liquid1. This indicates that more CO₂ exchange occurs at higher flow rates of the sweeping medium. As illustrated in Figure 5, pH increases of liquid1 versus operating time, which demonstrates the CO₂ removal affinities of air and water as sweeping media, pursue nearly the same trend at the same flow rates of sweeping media. However, each flow rate has an intersection point between air and water curves. In all cases, before this point, which means lower pH or higher aqueous CO₂ concentration, the affinity of air for CO₂ removal is greater than that of water. On the contrary, after the intersection point, this phenomenon becomes vice versa.

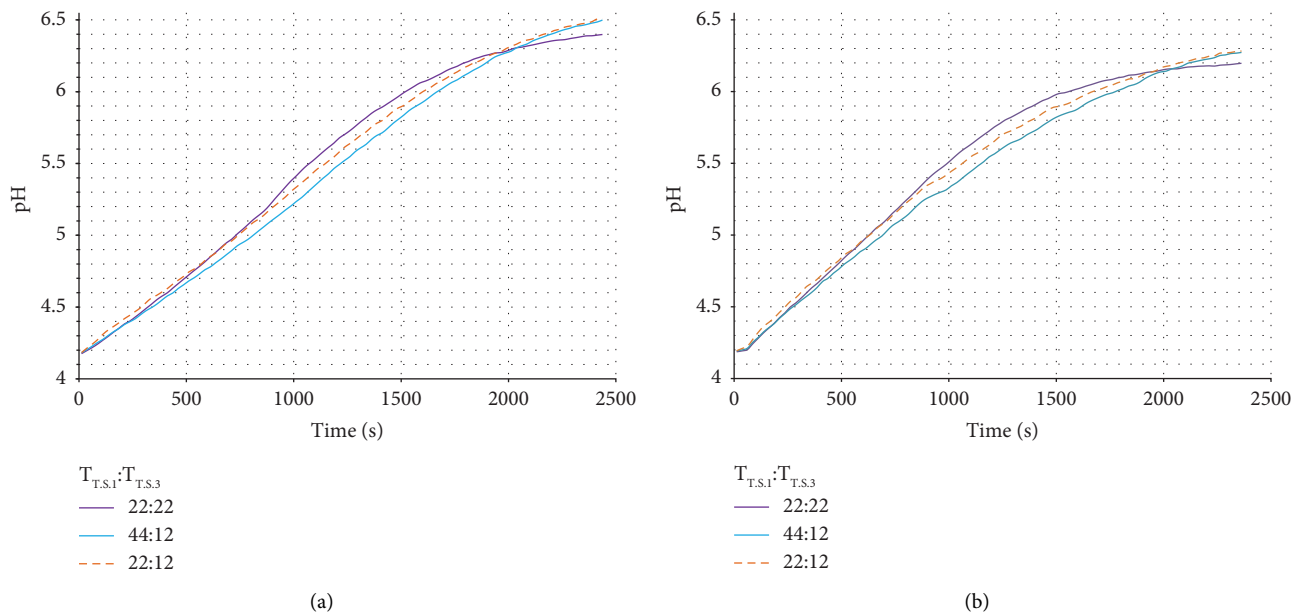
Moreover, as shown in Figure 5, the intersection points of water and air as CO₂ sweeping media appear in a lower pH by increasing their flow rates. In other words, it is observed that during aqueous CO₂ removal in higher flow rates of the sweeping medium, water becomes more efficient than air. This could be addressed by the effect of turbulency in enhancement of mass transfer capability of water. On the other hand, in higher CO₂ concentration, the diffusivity of carbon dioxide in air is more effective than its dissolution in water. Furthermore, it is observed that the plateau section of the curves, which its related pH represents the CO₂ removal capacity of the process, in the case of using water as sweeping media is higher in relation to the cases of using air instead.

TABLE 1: Temperature variations recorded by sensors 1–4 (mean \pm SD) in the case of using water as fluid2.

Test no.	Sensor				Average temperature between two sides of membrane at liquid 1 inlet; outlet ports ($^{\circ}$ C)
	$T_{T.S.1}$ ($^{\circ}$ C) (Liquid1 inlet)	$T_{T.S.2}$ ($^{\circ}$ C) (Liquid1 outlet)	$T_{T.S.3}$ ($^{\circ}$ C) (Water inlet)	$T_{T.S.4}$ ($^{\circ}$ C) (Water outlet)	
1	44.0 ± 0.10	36.4 ± 0.39	12.0 ± 0.01	43.2 ± 0.30	24.20; 43.60
2	22.0 ± 0.04	19.2 ± 0.33	12.0 ± 0.01	21.4 ± 0.44	15.60; 21.70
3	22.0 ± 0.05	21.9 ± 0.04	22.0 ± 0.06	22.1 ± 0.10	21.90; 22.00

TABLE 2: Temperature variations recorded by sensors 1–4 (mean \pm SD) in the case of using air as fluid2.

Test no.	Sensor				Average temperature between two sides of membrane at liquid 1 inlet; outlet ports ($^{\circ}$ C)
	$T_{T.S.1}$ ($^{\circ}$ C) (Liquid1 inlet)	$T_{T.S.2}$ ($^{\circ}$ C) (Liquid1 outlet)	$T_{T.S.3}$ ($^{\circ}$ C) (Air inlet)	$T_{T.S.4}$ ($^{\circ}$ C) (Air outlet)	
1	44.0 ± 0.12	38.2 ± 0.59	12.0 ± 0.01	43.1 ± 0.13	25.10; 43.50
2	22.0 ± 0.07	20.1 ± 0.23	12.0 ± 0.02	21.3 ± 0.38	16.10; 21.60
3	22.0 ± 0.05	21.9 ± 0.38	22.0 ± 0.01	21.9 ± 0.15	22.00; 21.90

FIGURE 6: Effect of temperature on aqueous CO₂ removal with DI water (a) and air (b) as the sweeping medium.

This difference can be originated from the effect of chemical dissolution in water case.

In Table 3, the intersection points of water and air, as the CO₂ sweeping media, are presented in mean \pm SD considering the repetition of experiments. In addition, the corresponding total CO₂ is calculated by the pH-based method [38] and shown in Table 3.

These results are obtained using one-pass air or pure water as a CO₂-extracting agent. A much higher difference in CO₂ removal capacity is expected by comparing them with the experiments implemented using diethanolamine as a sweeping or extracting agent [32].

It is noteworthy that counter-current one-pass stream of sweeping media increases the efficiency of the removal process by shortening the time needed to reach the plateau section (maximum removal capacity) because it is expected to have more overall mass transfer driving force and consequently more mass transfer rate. This can result in smaller required retention time.

As presented in Table 3, a higher flow rate (or less retention time) results in lower pH values and more concentration of CO₂ at intersection points, leading to less CO₂ removal at the intersection point. Also, the pH changes vs. time curves in Figure 5 indicate that there is a direct

TABLE 3: The average of intersection points (\pm SD) in each flow rate at 22°C.

Flow rate (ml/min)	500	750	1000	1500
Intersection pH	6.06 \pm 0.015	5.95 \pm 0.015	5.33 \pm 0.026	5.10 \pm 0.015
Corresponding total CO ₂ concentration (ml CO ₂ /l)	3.53 $\times 10^{-6}$ \pm 1.90 $\times 10^{-7}$	5.23 $\times 10^{-6}$ \pm 2.92 $\times 10^{-7}$	6.07 $\times 10^{-5}$ \pm 6.62 $\times 10^{-7}$	1.66 $\times 10^{-4}$ \pm 1.13 $\times 10^{-7}$

correlation between the flow rates and pH values, while the lower flow rates result in higher CO₂ concentration at a specific time.

It is believed that water with high temperature has more tendency to release its dissolved CO₂ than cold water regarding endothermicity of this phenomenon [42]. Since the CO₂ absorption process is exothermic and, in contrast, gas stripping is endothermic [43–45], it is expected that the temperature of liquid1, as the CO₂ carrying medium, should be increased, while the temperature of fluid2, as the CO₂ absorbing medium, should be decreased in order to transfer more CO₂ from liquid1 to fluid2. However, this expectation has not been yet properly scrutinized.

According to Figure 6, the trends of pH changes versus time in three different temperature ratios are similar for both liquid-liquid and air-liquid HFMCs. As it can be seen, two pH ranges could be discriminated. In both types of HFMCs, for pH values between circa 5.3 and 6.2 (i.e., total CO₂ concentration between 2.17×10^{-6} and 6.87×10^{-5} ml CO₂/l at 22°C), equality of temperatures of sweeping and carrying media at 22°C resulted in most efficiency. After this region, for pH values above 6.3 (i.e., total CO₂ concentration lower than 1.57×10^{-6} ml CO₂/l at 22°C), the equality of temperatures at 22°C led to least efficiency in both types of HFMCs, while the results for the two other temperature ratios were nearly identical. Our results reveal no clear conclusion for a pH range below 4.9 (i.e., total CO₂ concentration greater than 3.95×10^{-4} ml CO₂/l at 22°C).

As shown in Tables 1 and 2 for both cases of using water and air as sweeping media, while there had been a temperature difference between two inlets of HFMC, and this difference became almost nonexistent at the outlet. This shows a high capability of selected hollow-fiber membranes (here, micro-porous polypropylene) for conductive heat exchange too.

5. Conclusions

This study uses a practical HFMC to compare the removal of aqueous CO₂ between water and air as sweeping media. In addition to aqueous CO₂ concentration, the effects of flow rates and temperature ratios of the CO₂ carrier and sweeper media on the CO₂ exchange rate were examined. The findings show that the maximum pH difference between the water and air graphs becomes minor at larger flow rates of sweeping media. This has the practical effect of allowing air and water to be utilized interchangeably as sweeping media for aqueous CO₂ removal in HFMCs. Aqueous CO₂ removal from water would typically be more effective than air at lower flow rates. According to the crossing point of the water and air curves for each identical flow rate, there is a certain CO₂ concentration at which the affinities of water and air for aqueous CO₂ removal switch from positive to negative. Interestingly, when the sweeping media flow rate increases, the pH for this crossing point decreases. After this intersection point, i.e., lower aqueous CO₂ concentration, water would be a more efficient CO₂ absorbent than air.

The results indicate that the temperature similarity of the two fluids in both types of HFMCs, i.e., liquid-liquid and

gas-liquid, is the most preferred condition to remove aqueous CO₂. However, at very low CO₂ concentrations, such as lower than 1.57×10^{-6} ml CO₂/l, cooling sweeping media and creating a temperature gradient between the two phases could remove more aqueous CO₂. The HFMC's design may be altered to employ the ideal sweeping medium, resulting in the intended CO₂ removal, depending on the intended use and operating conditions. The accurate removal of a patient's blood's CO₂ content is critical in several challenging therapeutic applications such as AVCO2R and VVCO2R. As a result, this research could be used as a foundation for improving such treatment techniques and enhancing patient improvement.

In conclusion, this study indicates that both air and water have comparable abilities to remove aqueous CO₂. As a sweeping medium, in higher CO₂ concentration, the affinity of air for CO₂ removal is greater than water. For further investigations, the effect of different sweeping media and flow regimen on aqueous CO₂ removal can be studied. The design of the HFMC can be modified to obtain higher contact surface and better CO₂ removal as a result. Moreover, the functionality of the proposed HFMC at low pH (lower than 4.9) is well determined.

Data Availability

All data needed for the reproducibility of this research are adequately provided within the article. The data that support the findings of this study can be made available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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