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ABSTRACT

SIMULTANEOUS HEAT AND MASS TRANSFER TO A LIQUID THROUGH TWO SEPARATED INTERFACES

by
Ku-il Park

The oxygenator is the main element of a heart-lung machine, which takes over the work of the lungs (adding oxygen to and removing carbon dioxide from the blood) during an operation. Oxygenators function by passing oxygen through a hollow fiber (straw-like fiber), where blood gases are exchanged via tiny pores in the fiber walls. In this study, a fiber-in-fiber technology was used to design a temperature controlled blood oxygenator membrane module. A Celgard® X-10 hollow fiber was put through the lumen of a polypropylene hollow fiber having a solid wall. Both fibers are made of polypropylene and are waterproof due to their hydrophobic properties. A heating fluid (e.g., deionized water) was circulated on the shell side of the outer set of the nonporous hollow fiber having a solid wall. In this way, the temperature level of the oxygenated deionized (DI) water was maintained at the desired temperature. The hollow fiber module carried out the removal of carbon dioxide and absorption of the oxygen under various conditions of temperature and flow rate. The hollow fiber module, which was used for this experimental work, was effective in removing carbon dioxide. High percentage removal of carbon dioxide was achieved at lower feed water flow rates while oxygen was absorbed into the water stream. The flow rate of the feed water and the temperature of the circulated water were important factors controlling the concentration of dissolved oxygen in water.

SIMULTANEOUS HEAT AND MASS TRANSFER TO A LIQUID THROUGH TWO SEPARATED INTERFACES

by

Ku-il Park

A Thesis
Submitted to the Faculty of
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In Partial Fulfillment of the Requirements for the Degree of
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APPROVAL PAGE

SIMULTANEOUS HEAT AND MASS TRANSFER TO A LIQUID THROUGH TWO SEPARATED INTERFACES

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This thesis is dedicated to my parents,

Kwan-hwa Park and Hee-hyuck Lim.

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CHAPTER 1

INTRODUCTION

In artificial organs as well as chemical equipments, one often needs to carry out mass transfer as well as heat exchange. Conventionally, these two processes are usually carried out sequentially, for example, mass transfer followed by heat exchange or heat exchange followed by mass transfer. (There may be occasions where it would be beneficial to have this carried out simultaneously). If one is to carry out these processes simultaneously, one has to have two interfaces, the mass transfer interface and the heat transfer interface. In some chemical engineering devices based on dispersion of one phase in another for mass transfer, this would be carried out as follows. Oxygen to be absorbed in a liquid in a vessel would be bubbled through the liquid by a sparger; there would be separate cooling or heating coils in the vessel to cool or heat the liquid [1].

In artificial organs employed for various treatments for blood, oxygen bubbling is avoided. Commonly porous hydrophobic hollow fibers are used to oxygenate blood in blood oxygenators. In liver assist devices, the blood to be treated by hepatic cells has to have a high oxygen concentration. Blood taken out of the patient has also to be heated up to 36.5°C for this purpose before it is introduced to the region containing the hepatocytes [2]. Both of these functions may be carried out in one device if an appropriate hollow fiber based design could be developed.

In this thesis, to achieve these goals, a device has been developed pursuing the suggestion by Professor K.K.Sirkar. As shown in Figure 1.1 and Figure 1.2, this device essentially is like a double-pipe heat exchanger except the inner tube is a hydrophobic

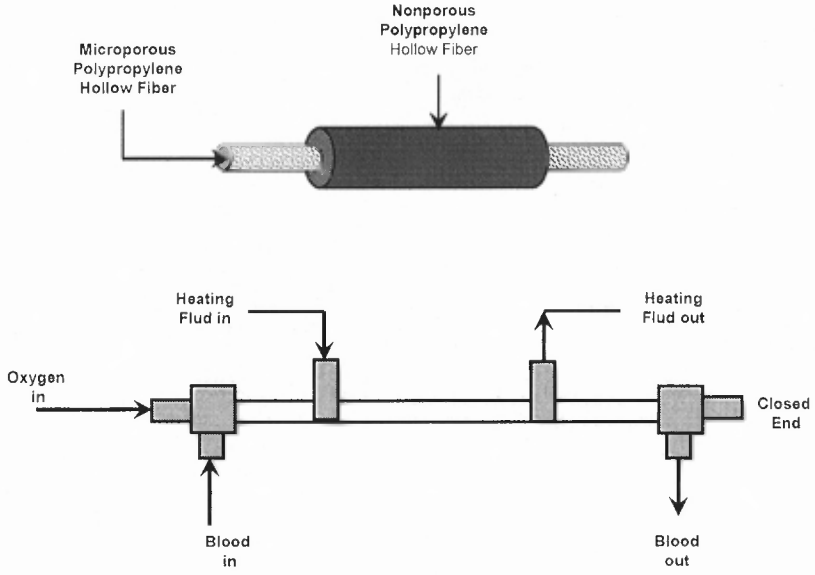


Figure 1.1 Schematic of the Fiber-in-fiber Hollow Fiber Module.

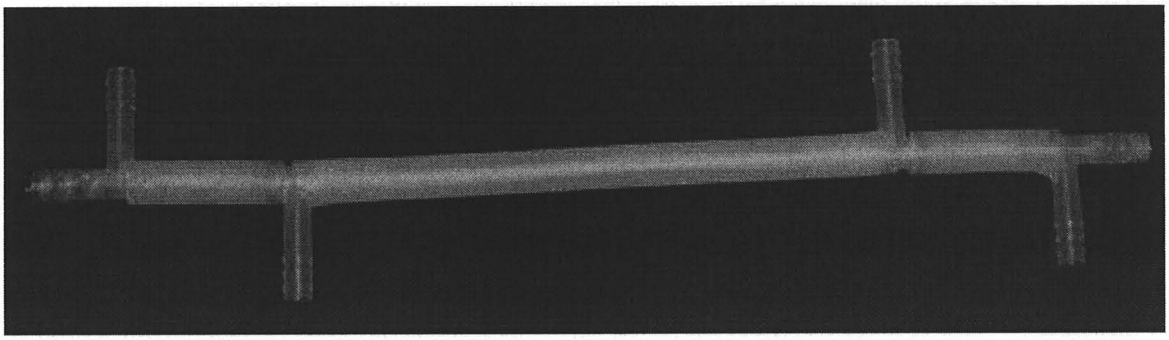


Figure 1.2 Photograph of the Fiber-in-fiber Hollow Fiber Module.

microporous hollow fiber. The shell side of the outer tube having a solid nonporous wall has hot water flowing. The annulus has deionized water (representing blood) flowing. The lumen of the porous capillaries in the double-pipe device has oxygen flowing through it. Thus the water (i.e. , blood) flowing in the annulus may be heated (or cooled) as needed; simultaneously through the other gas-liquid interface on the outside diameter of the inner hydrophobic porous hollow fiber, oxygen could be introduced into the water (i.e. , the blood phase) and carbon dioxide could be stripped from the water into the gas phase (if the gas phase flows out of the fibers unlike that shown in Figure 1.1).

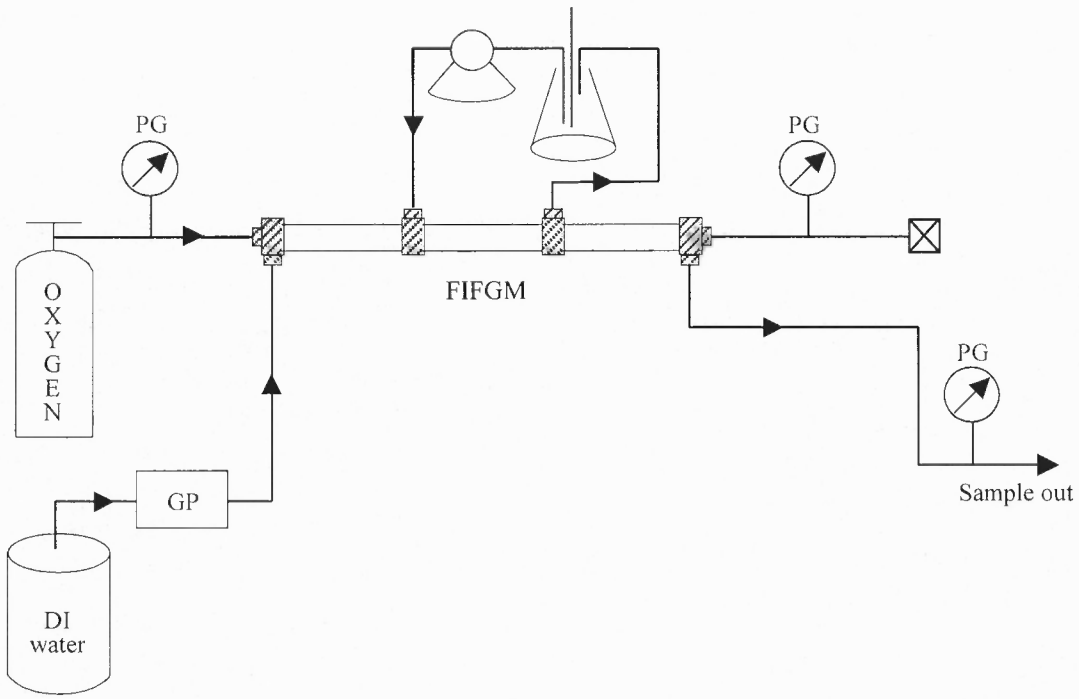
CHAPTER 2

EXPERIMENTAL

2.1 Fiber-in-fiber Blood Oxygenator Membrane Device and Experimental Setup

2.1.1 Membrane Device Description

Preliminary blood oxygenator experiments were carried out with the setup shown in Figure 2.1. In this study, fiber-in-fiber technology was used to design a temperature controlled blood oxygenator membrane module. Module 1 (Figure 1.1) consists of two parallel hollow fiber bundles, where each end of each bundle is independent of the other. One bundle e.g., the inner hollow fiber bundle consisting of fibers having a smaller OD/ID was such that each such fiber was coaxially placed inside the inside diameter of individual fibers in the second bundle e.g., the outer bundle of hollow fibers having an ID larger than the OD of the smaller fibers. Once the smaller ID fibers are placed within the larger ID fibers, the composite bundle of hollow fiber-in-fiber is encased in a tubular housing with an inlet and an outlet port with each bundle independently sealed at each end. A picture of the module is provided in Figure 1.2. This unique hollow fiber device is designed in such a way, where one set of Celgard X-10 microporous polypropylene hollow fiber (**ID/OD: 110/160μm**:, Hoechst-Celanese, Charlotte, North Carolina) was inserted in the lumen of a second set of nonporous solid wall polypropylene hollow fibers **(ID/OD: 425/575μm, Celgard, Charlotte, NC)** (Figure 1.1). Detailed characteristics of the fiber-in-fiber hollow fiber membrane module (FIFHFM) are listed in Table 2.1.



GP= Gear Pump

PG= Pressure Gauge

FIFGM= Fiber-In-Fiber Gasification Module

Figure 2.1 Experimental Setup for Blood Oxygenator Membrane Process.

Table 2.1 Specification of the Fiber-in-fiber Module and Fibers

SPECIFICATION OF FIBERS

Solid Hollow Fiber Type	= CELGARD
Porous Hollow fiber type	= CELGARD® X-10
Material of Hollow fiber	= Hydrophobic Polypropylene
X-10 Fiber Inner Diameter	= 110 μm
X-10 Fiber Outer Diameter	= 160 μm
Solid Fiber Inner Diameter	= 425 μm
Solid Fiber Outer Diameter	= 575 μm
Solid Fiber Wall Thickness	= 75μm
Pore size in Porous Fiber Wall	= 0.04 μm x 0.011μm

SPECIFICATION OF MODULE

Length of Module	= 13.75 inch
Diameter of Module	= 0.53 inch
Number of Fibers of each type	= 10
Tube type	= Low Density Polyethylene

Total fiber surface area based on inner fiber O.D = 2.01×10^5 μm^2

Total fiber surface area based on outer fiber I.D = 2.5954×10^6 μm^2

2.1.2 Experimental Setup (Oxygenating DI Water)

The setup used to carry out the initial experimental work is described in Figure 2.1. A pure oxygen gas stream was fed through the lumen of the inner set of the microporous hydrophobic hollow fibers, while the outlet end was closed off. A pressure gauge (Matheson, E. Rutherford, NJ) at the outlet of the module indicated the gas pressure. Fresh deionized (DI) water was pumped through the annulus (e.g., ring like space between the inner and the outer hollow fiber) using a peristaltic pump (Masterflex, Cole-Parmer, Vernon Hills, Illinois). The pressure of the DI water in the annulus was maintained 5-10 psi higher than the gas pressure in the bore of the inner bundle hollow fibers to prevent the bubbling of the O_2 into the liquid in the annulus.

Oxygen absorption took place at the gas-liquid interface maintained at the pore mouths of the inner hollow fiber into the liquid side without wetting the membrane. This method of operation is known as the nonwetted mode of operation since the liquid does not wet the membrane pores as they are gas filled [3]. A pressure gauge on the liquid side (Matheson, E. Rutherford, NJ) was used to monitor the annulus DI-water pressure. While the oxygen rich water was passed through the annulus, a heating fluid (e.g., deionized water) was circulated on the shell side of the outer set of the nonporous hollow fiber using a circulator pump from a temperature controlled bath (Model 002-4175, Hakke, Karlsruhe, Germany). In this way, the temperature level of the oxygenated DI water was maintained from 28°C to 38°C. The temperature level was maintained via the circulator pump from the temperature controlled bath (Model 002-4175, Hakke, Karlsruhe, Germany) and it was measured with a thermometer.

During this process, pure oxygen diffuses across the inner microporous hollow fiber

membrane into the DI water which is flowing through the annulus [4]. The concentration of dissolved oxygen in the annulus-side DI water at the outlet end was measured by a Polarographic Dissolved Oxygen Electrode (Model 830A, Orion Research, Beverly, MA) at a certain temperature level. The temperature level was measured and recorded for each experimental run. Dissolved oxygen concentrations in DI water was monitored at the inlet and the outlet of the membrane module by drawing samples from the process line. The measurement range of this electrode was 0-60mg/l or ppm at 20°C. The electrode was calibrated every other day using 2-point calibration which are air and liquid calibration. The pure DI water flow rate in the annulus was varied from 25ml/min to 150ml/min. Another experiment was carried out by increasing the temperature of the circulating water from 42°C to 52°C in order to compare the DO of the sample at each flow rate.

2.2 Oxygen Degasification and Oxygenation

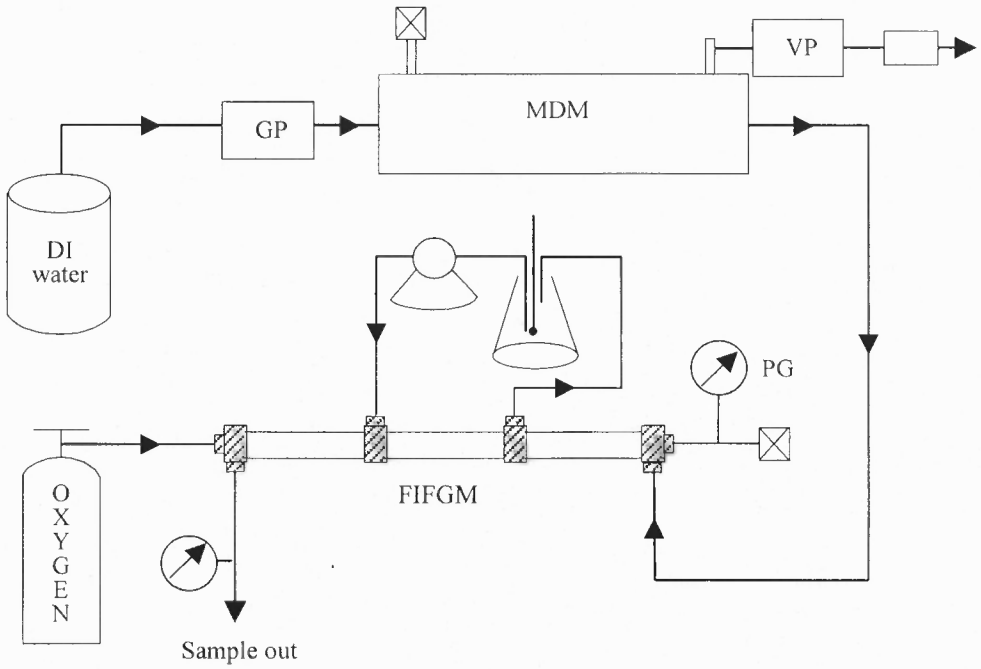
In order to achieve maximum absorption of oxygen, another module (Liqui-Cel™, Hoechst Celanese, Charlotte, NC) was prepared to remove any dissolved gas. Specification of such a hollow fiber module is provided in Table 2.2. The DI water stream was fed through the bore side of the microporous hydrophobic fibers, and the line was connected to the fiber-in-fiber absorption module. Vacuum was applied to the shell side of the module with the other end closed.

Table 2.2 Specification of Hollow Fiber Module.

Module (Name)	Manufacturer	Type of Hollow Fiber	Flow configuration	Overall size	Number of fibers	Fiber i.d / o.d (cm)	Mass transfer area (cm^2)	Description of Process Studied
Liqui-Cel	Hoechst-Celanese	Microporous Polypropylene, Celgard X-10	Parallel flow	2"x 10"	7,500	0.024/0.03	17,000	Absorption

2.2.1 Experimental setup for degasification and oxygenation of DI water

The experimental setup used for this study is shown in Figure 2.2. This experimental setup used two membrane units. The first module (e.g., degasification module) was used for gas removal; the second module was used for gas absorption (absorption module). The aqueous stream (e.g., DI water) was initially pumped through the bore of the degasification module using a Masterflex pump (Cole-Parmer, Vernon Hills, Illinois), while vacuum (KNF Neuberger, Laboport, Beverly, MA) was pulled on the shell side of the module in order to create a driving force for oxygen removal. The degasification module was obtained from Hoechst Celanese and had microporous polypropylene, Celgard X-10 hollow fibers; the fiber-in-fiber absorption module was made in the laboratory as described in section 2.4. At the outlet of the degasification module a liquid sample was drawn to check the oxygen concentration in the outlet stream of the degasification module. The oxygen concentration was measured using an oxygen probe (Model 830A, Orion research, Beverly, MA). Degassed aqueous stream was fed into the bore side of the microporous hollow fiber in the absorption module. Sample analysis and experimental procedure for the absorption module were previously described in section 2.1.2.



GP= gear Pump
PG= Pressure Gauge
VP= vacuum Pump
MDM= Membrane Degasification Module
FIFGM= Fiber-In-Fiber Gasification Module

Figure2.2 Experimental Setup for Degasification and Oxygenation of DI Water.

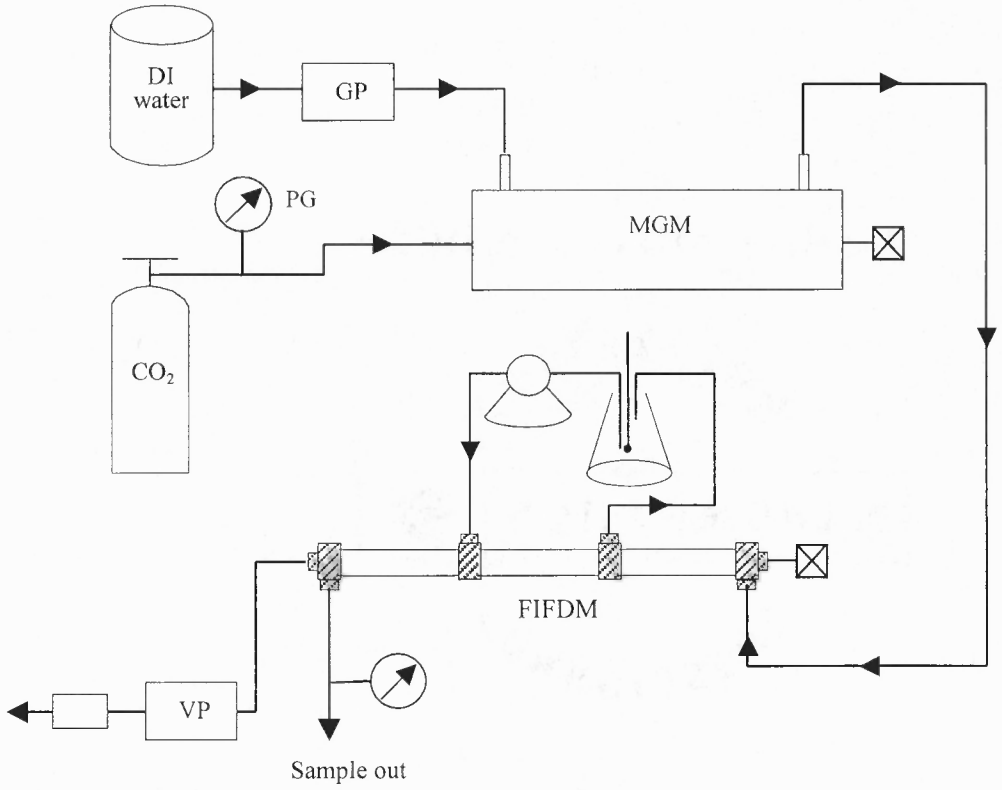
2.3 Carbon Dioxide Degasification

Degasification experiments for removing gas (carbon dioxide) from DI water were carried out using a blood oxygenator membrane module. Table 2.1 provides the details of the membrane module. To carry out stripping, the water needed to be carbonated first. A hollow fiber module was employed to dissolve pure gas (carbon dioxide) into a DI water stream for preparing the feed stream for the degasifier. Specification of the hollow fiber module is provided in Table 2.2.

2.3.1 Experimental Setup for Degasification of Carbon Dioxide

The experimental setup and its photo are shown in Figure 2.3 and Figure 2.4 respectively. The absorption module (Liqui-Cel™, Hoechst Celanese, Charlotte, N.C) was used for preparing the water saturated with carbon dioxide. The gas (carbon dioxide) was supplied to the bore of the fibers in the absorption module with outlet end closed while DI water was pumped through the shell side of the module by using a peristaltic pump (Masterflex®, Cole-Parmer, Vernon Hills, Illinois). A pressure gauge (Matheson, E. Rutherford, NJ) at the outlet of the module indicated the gas pressure. The pressure of the DI water in the shell side was maintained 5-10 psi higher than the gas pressure in the bore of the inner bundle of hollow fibers to prevent the bubbling of the CO_2 into the liquid in the annulus [5]. A pressure gauge at the liquid side (Matheson, E. Rutherford, NJ) was used to monitor the shell side DI-water pressure. A sample was drawn from the end of the module by a bypass line in order to measure the concentration of carbon dioxide in the outlet stream from the absorption module.

The carbon dioxide concentration was measured using a CO_2 electrode (Model 95-02, Orion Research, Beverly, MA). The measurement range for this electrode was 4.4 to 440ppm. As the absorption of CO_2 in water is essentially physical, the dissolved carbon dioxide concentration measurement procedure requires immediate buffering of the samples to avoid the escape of CO_2 from the liquid phase. A 50ml volume of carbonated water was collected from the above bypass line into a graduated cylinder, and 5ml volume of buffer liquid was pipetted simultaneously into this water. The dissolved CO_2 probe was immersed into the sample immediately, and a reading of the concentration in ppm was displayed in a digital read-out meter(290A, Orion research, Beverly, MA). The electrode was calibrated every other day using a 5-point calibration method. (A similar procedure was followed for the measurement of the dissolved carbon dioxide concentration in the sample obtained from the end of degasification module.) In the degasification module, the stream from the gas absorption module was passed through the annulus while vacuum (KNF Neuberger, Beverly, MA) was pulled on the bore side of the inner fibers of the module in order to create a driving force for carbon dioxide removal. In order to keep the temperature level of DI water saturated with carbon dioxide, heated water was circulated on the shell side of the degasification module.



GP= Gear Pump
VP= Vacuum Pump
PG= Pressure Gauge
MGM= Membrane Gasification Module
FIFDM= Fiber-In-Fiber Degasification Module

Figure 2.3 Experimental Setup for Degasification of Carbon Dioxide

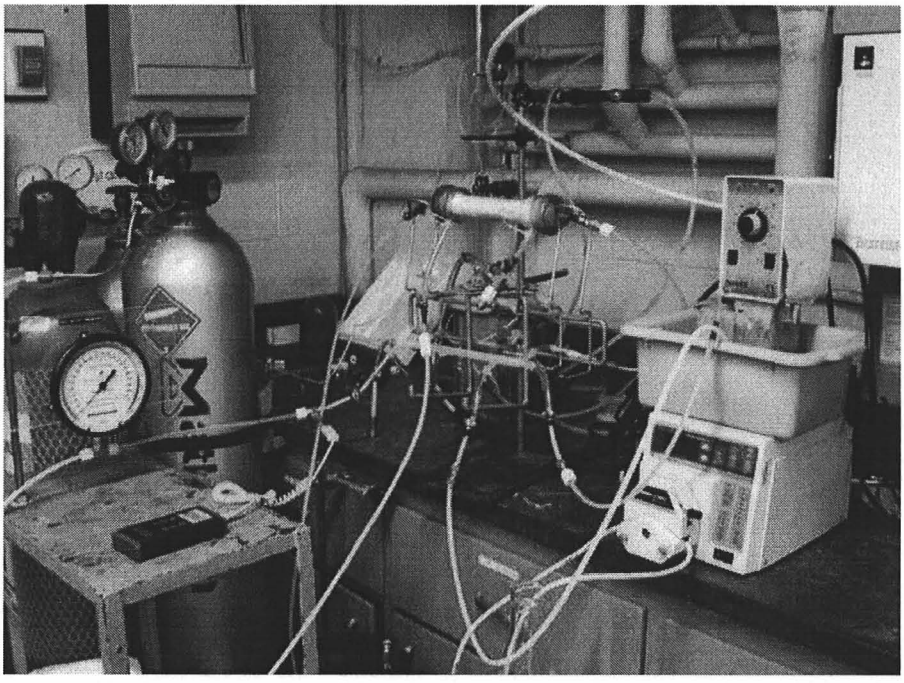


Figure 2.4 Photograph of the Experimental Setup.

2.4 Fabrication of the Fiber in Fiber Hollow fiber Module

In order to carry out these experiments, a module containing 10 hollow fibers each of which has a smaller microporous hollow fiber inside and 4 connectors (2 T-connectors, 2 cross connectors) was built (Figure 1.1). The first step in module making was cutting the solid fibers (ID: 425μm; OD: 575μm) to the desired length. Each end of the solid fiber was treated with chromic acid for 5 minutes to increase surface adhesion of the epoxy during potting. A Tubing (LDPE 0.375 x ½") was prepared in order to take 10 solid fibers inside the tube. A silicone rubber adhesive (RTV11, General Electric, Waterford, NY) was used to pot both ends of the solid fiber against the internal diameter of the tube. This layer of potting was necessary because epoxy does not make good bonding with fibers. After solidifying the silicone rubber adhesive for one day, epoxy was poured to the desired place and cured for a day. The epoxy resin mixture with 4:1 ratio of C-4 resin to activator D (Beacon Chemical Co., Mt Vernon, NY) was used for sealing. The epoxy mixture was exposed to vacuum for a short time to remove any gases from the mixture. This layer was also poured into the gap between connector and tube. After complete solidification of epoxy, each of the ten X-10 fibers was inserted through the bore of each hollow solid fiber; both ends of these fibers sticking out were sealed with silicone rubber and epoxy into separate connectors in the manner described earlier.

After potting was finished and the epoxy layers were completely cured, the module was tested for leakage. The shell side was filled with deionized water and then pressurized with nitrogen while the other end was closed. The pressure was increased slowly up to 20 psig. If no water came through the tube side, this module was indeed found to be leak-free vis-à-vis the solid fibers. The same procedure was followed by introducing water

into the annulus through one end while the other end was closed. Then this water was pressurized; if no water came out through the tube side or the fittings, then it was leak free.

CHAPTER 3

3. Results and discussion

3.1 Oxygenating DI Water with Fiber-in-fiber Blood Oxygenator

The results of oxygenating DI water with the fiber-in fiber hollow fiber module are presented in Table 3.1. As shown in Table 3.1, DO of the sample from the liquid exiting the device had a large difference between the two flow rates 25ml/min and 50ml/min compared to the difference between other flow rates due to the limited surface area of the fibers. As the flow rate was changed from 25ml/min to 150ml/min the DO of the sample decreased from 4.8mg/l to 2.5mg/l. The temperature of the sample was also decreased from 37.8°C to 28.8°C. Detailed conditions of the experiments and the Reynolds numbers for each flow rate are shown in the Figure 3.1 and Table 3.2. At the higher temperature of the circulating water, as Table 3.3 shows, a higher DO of the sample was achieved at the same flow rate which is also illustrated in Figure 3.2.

Table 3.1 Experimental Results for Oxygenating DI Water.

Flow rate of DI water(cc/min)	DO of outlet sample (ppm)	Temp. of outlet sample(° C)
25	4.8	37.8
50	3.3	34.3
75	3.1	32.0
100	2.9	30.6
125	2.7	29.5
150	2.5	28.8

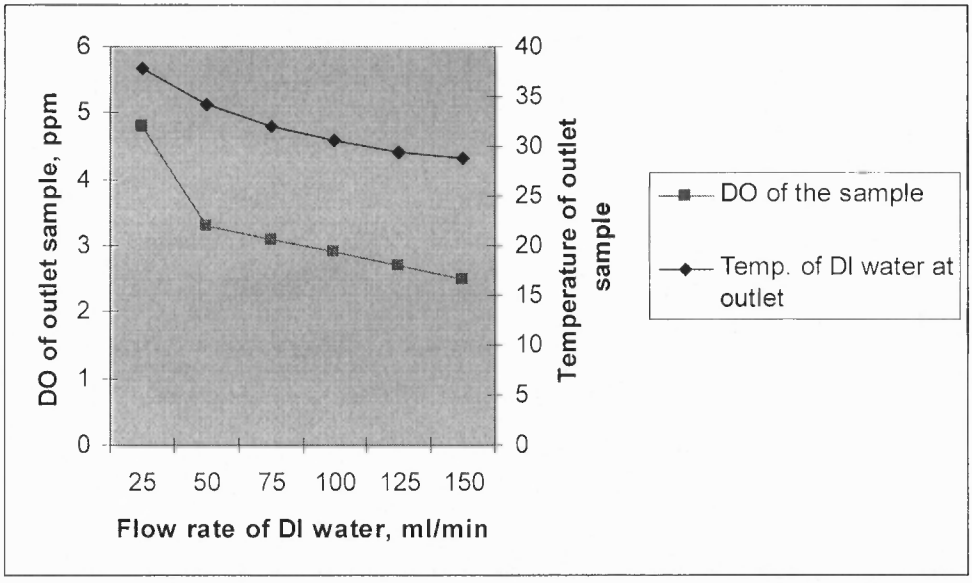


Figure 3.1 The Temperature and DO of Water at Outlet as a Function of Water Flow Rate.

Temperature and DO of feed DI water: 22.9° C, 2.0mg/l

Temperature of the circulating water at inlet: 42.5° C

Temperature of the circulating water at outlet: 41.1° C

Flow rate of the circulating water: 1600 ml/min

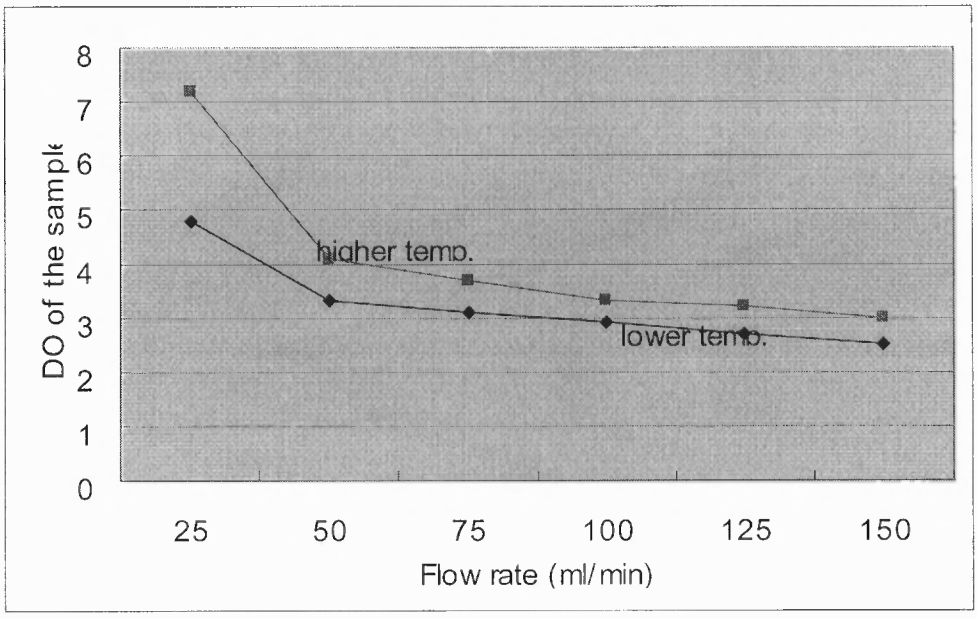


Figure 3.2 Experimental Results for the DO of Samples at Different Temperatures of Circulating Water: Temperature and DO of Feed DI Water: 23.7° C, 2.3mg/l

Table 3.2 Reynolds Number at Each Flow Rate.

Flow rate(ml/min)	Reynolds Number
25	647
50	1292
75	1945
100	2584
125	3237
150	3891

$$Reynolds\ \ number = \frac{velocity \times hydraulic\ \ diameter \times density}{viscosity}$$

$$hydraulic\ \ diameter = 4 \times \frac{\frac{\pi}{4}\left(D.I^2 - d_o{}^2\right)}{\pi(d_o + D.I)} = D.I - d_o$$

$$Flow\ \ cross-sectional\ \ area = \frac{\pi \times \left(D.I^2 - d_o{}^2\right)}{4}$$

$$velocity = \frac{volumetic\ \ flow\ \ rate\ (cc/min)}{flow\ \ cross-sectional\ \ area}$$

Table 3.3 Relationship between DO and Temperature of the Circulating Water.

Flow rate of DI water(cc/min)	DO at 42° C of circulating water	DO at 52° C of circulating water
25	4.8	7.2
50	3.3	4.1
75	3.1	3.7
100	2.9	3.3
125	2.7	3.2
150	2.5	3.0

3.2 Oxygen Degasification and Oxygenation

The concentration of the oxygen in the stream, which was passed through the degasification module, was 0.3mg/l at 22.3 ° C. The experimental results are shown in Table 3.3 and Figure 3.3.

3.3 Carbon Dioxide Degasification

The results of carbon dioxide degasification studies are presented in Table 3.5. The feed water stream had flow rates of 25-150ml/min and each data point in the plot (Figure 3.4) was taken after steady state. The percentages of overall CO_2 removals were ~100% to 85% for flow rates of 25-150ml/min of the feed water having 427-438 ppm CO_2. Almost complete removal of CO_2 was achieved from water streams flowing at 25-50 ml/min and having 438-432 ppm CO_2 in the feed water.

Table 3.4 Experimental Results of Oxygenating Degasified Water.

Flow rate of DI water(cc/min)	DO of outlet sample (ppm)	Temp. of outlet sample(° C)
25	4.7	28.7
50	2.6	28.5
75	1.9	27.7
100	1.7	27.2
125	1.6	27.0
150	1.6	26.9

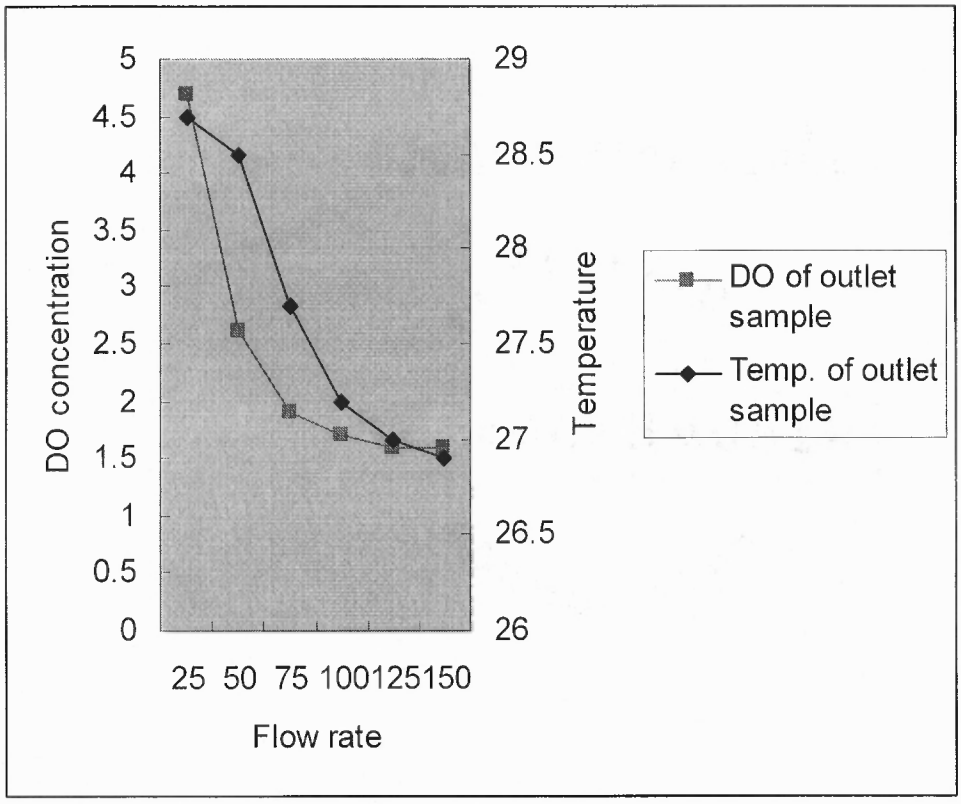


Figure 3.3 Experimental Results for Oxygenating Degasified Water.

Table 3.5 The Removal of Carbon Dioxide.

Flow rate of DI water(cc/min)	CO_2 concentration in the feed water (ppm)	CO_2 concentration in the outlet (ppm)
25	438	2.4
50	432	9.7
75	436	17.2
100	429	31.4
125	431	48.7
150	427	64.7

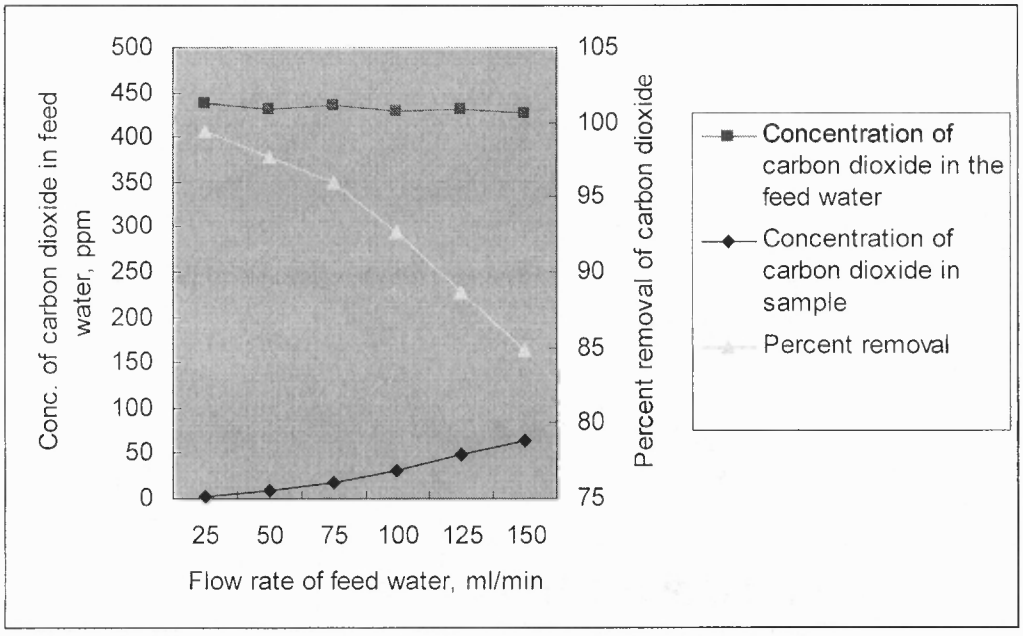


Figure 3.4 Percent Removal of Carbon Dioxide from Carbonated Water by Degasification with FIFDM.

CHAPTER 4

CONCLUSIONS

Processes based on the Fiber-in-fiber Hollow Fiber Membrane (FIFHFM) were successful in achieving transfer of oxygen into and removal of carbon dioxide from water as the temperature of the water was increased by heating. X-10 fibers put into the bore of each of solid fibers were found to perform effectively during the experimental work. Temperature controlled blood oxygenation could be easily achieved in one device using Fiber-in-fiber Technology.

The performance of the above-mentioned processes strongly depends on the flow rate of feed water and the temperature of circulating water. Efficiency of the FIFHFM process increases with decreasing flow rates of feed water and increasing temperature of the circulating water. To maintain the high efficiency of oxygenating process feed flow rates should be moderate (~25ml/min) with high temperature of circulating water. Higher feed flow rate and low temperature of circulating water leads to lower absorption of oxygen. High percent removal (~97-100%) of carbon dioxide was also achieved at lower flow rate of feed water (~25-50ml/min).

The Fiber-in-fiber Technology mentioned above may stimulate new types of blood oxygenators due to the compactness of the device, being easy to handle and design.

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