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Fabrication of Tri-bore Hollow Fiber (TBF) Membrane Module for Vacuum Membrane Distillation

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Abstract: Highly constrained by the requirements of high porosity and micropore sizes, during long term operations, the traditional single-bore hollow fiber membranes have several problems; among them, the easy breakage and performance instability. In this work, Tri Bore Hollow Fiber (TBHF) membrane with triangle outer geometry was designed and successfully fabricated via the specially designed spinneret and optimized spinning conditions. TBF module is also prepared and manufactured in the easiest way possible. Besides, the TBF membranes exhibited superior stability in terms of vapor permeation flux and salt rejection during the MD experiment with robust operational conditions.

Keywords: Tri- bore, Hollow Fiber, TBHF module, desalination, Membrane Distillation.

1 Introduction

Freshwater shortages and water scarcity are major global issues, especially in the arid and semiarid regions of the world. Desalination id progressively implemented over the last decades as an alternative to providing pure water [1–5].

Globally, more than 80% of the world's desalination capacity is provided by two techniques: thermal (Multi-Stage Flash (MSF) distillation, Multi-Effect Evaporators, Mechanical, and Thermal Vapor Compressions), and membrane (Reverse Osmosis (RO), Nanofiltration, Electro Dialysis [6], and Membrane Distillation (MD)). The Membrane Distillation (MD) is a new emerging technology, which has the advantage of performing at moderate temperatures and pressure [2, 3, 7, 8]. It is economical in terms of energy because the heat source for the process can be low-grade energy sources [2, 8, 9, 10, 11]. MD has four configurations based on the way of vapor collection: (a) Direct Contact MD (DCMD), (b) Air Gap MD (AGMD), (c) Sweeping Gas MD (SGMD), and (d) Vacuum MD (VMD) [2,9,10,12,13].

Flat sheet and Hollow Fiber (HF) membranes are widely used in water desalination, the HF membranes have essentially the following advantages: (1) a much greater membrane area per unit volume of the membrane module, (2) self-mechanical support, and (3) good flexibility [14].

Since Single Bore Hollow Fiber (SBHF) membrane has modest mechanical strengths, it may break easily during use. Besides, polymeric hollow fibers may elongate, twist, stretch, curl, or wrapped with one another, thus, creating difficulties during daily operation or in back-washing for fouling removal [15]. SBHF membrane has easy installation, simple processing, and so on, but, the main disadvantage of the SBHF membrane is the poor mechanical properties, as well as, the low strength versus the required high pressure. Therefore, the novel membrane of Multi Bore Hollow Fiber (MBHF) membranes leads to improve the mechanical properties, which could combine the characteristics of flat sheet and hollow fiber membranes to give the advantages of both. The lotus root in nature exhibits an exceptional geometry with consistently axially aligned hollow channels. This structure affords one of the best membrane geometries, which promises large porosity, enhances hydrodynamics

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performance, and creates excellent mechanical strength. The scientists have been elaborate to mimic the lotus-root structure to attain micro or nano-structure with both high mechanical strength and the large surface area or sorption properties [16]. The merits of the MBHF membrane are very promising, where a 75% reduction in energy consumption was achieved, as compared with that of SBHF membranes. However, using a membrane with Multi Bore Hollow Fibers (MBHF) is a promising opportunity, where the bores are homogeneous, aligned, cylindrical, which will reflect on the membrane tortuosity, and lead to a negligible hydraulic resistance.

In recent years, the Multi Bore Hollow Fiber (MBHF) membrane has attracted excellent attention because it is durable and has not suffered from fiber cracking as SBHF Membrane. They apply in all kinds of membrane separation. Rectangular with Five Bore, circular Seven, Five and Four Bore and Triangular of Three Bore of PES are earlier prepared with a wet-spinning method [17], they have high mechanical strength on account of the Multi Bore supporting structure.

In recent years, many researchers have made great efforts to improve the mechanical strength of Membrane Distillation HF membranes [18-20]. In this respect, the MBHF membrane is considered the best candidate, which ensures both large porosity and excellent mechanical strength. It successfully improves tensile strength in both axial and radial directions [21, 22].

MBHF membranes are commercialized; the following companies are the pioneers: (1) Multi Bores polyethersulfone (PES) HF membranes are consisting of seven bores developed by **Inge GmbH**. (2) Tri Bore (Triangle) PES or polyvinylidene fluoride (PVDF) HF membranes launched by **Hyflux**.

Both of these membranes offered excellent durability and mechanical strength while maintaining the same permeate production rate as compared with the SBHF membranes. A series of MBHF membranes with various bores configurations and pore sizes for UF, membrane distillation (MD), and Forward Osmosis (FO) applications are commercialized. The MBHFs for MD is not only showed high permeation fluxes and energy efficiency but also, they possess superior stability and robustness.

Because of the aforementioned advantages of MBHF membranes, this article aims to design and demonstrate a simple way to fabricate a lab-scale TBHF membrane module using manufactured available parts and detailed procedure, it will step wisely describe the potting process. A most suitable procedure for the subsequent fabrication of TBHF membranes is identified, based on the MD performance, it is studied for various flat sheet membranes. Afterward, the TBHF module was fabricated, and their performance for MD was systematically investigated. The outcome of this study may provide useful insights towards the design of next-generation HF membranes for membrane distillation applications.

The current article is aiming at a design and exhibition of a simple method to manufacture a lab-scale TBHF membrane module with detailed procedures. The suitable procedures for the fabrication of TBHF membranes are evaluated. The TBHF module was fabricated, and their performance for MD was investigated. The outcome of this study may provide valuable insights into the design of next-generation HF membranes.

2 Materials and Methods

2.1 Materials

A commercial PVDF powder and n-Methyl-2-pyrrolidone (NMP) were provided by Alfa Aesar Inc. and ROTH respectively. Ethylene Glycol (EG) used in polymer preparation were purchased from Acros Organics. Lithium chloride (LiCl) with a purity of 99% and Aluminum silicate (AL2SiO5) powder were, also, provided by Alpha Chemical and PANREAC QP respectively.

2.2 Membrane Preparation

2.2.1 Flat sheet Membrane Development

PVDF flat-sheet membranes were prepared and compared to its performance and determine the most suitable for TBHF membranes; they are prepared by phase inversion via immersion precipitation method. The dope solution was prepared by dissolving PVDF polymer in NMP with the addition of EG and LiCl as a pore former in the solution at around 50 °C while stirring mechanically at 400 rpm for 4 h. The dope solution was continuously stirred at low speed to prevent any sedimentation. It was degassed overnight. Some modification was achieved using the addition of the Aluminum silicate with different concentration (2-5%). The solution was cast on to a glass plate at ambient temperature, then the film immersed at room temperature in a rectangular coagulation bath (water). Different blends were prepared as depicted in the Table (1).

Table 1: Weight ratios of the polymer and additives used to prepare the dope solution.

Name of dope solution	Composition%					
	PVDF	EG	LiCl	NMP	TEOS	AL_2SiO_5
P1	13	5	5	77	-	-
P1A 5%	13	5	5	72	-	5
P1A 2%	13	5	5	75	-	2
P1T	13	5	5	72	5	-

These prepared flat sheet membranes were characterized, and also, the contact angle of these membranes was determined. The best obtained membrane, which will give the suitable porosity and hydrophobicity, was used to spin the multi bore hollow fiber membranes.

2.2.2 Spinneret Design and Fiber Fabrication

TBHF membranes of triangle outer geometry were fabricated via phase inversion spinning process using a specially designed three-needle spinneret, as shown in Figure (1). The schematic drawing of the spinneret and coagulation bath were presented in Figure (2).

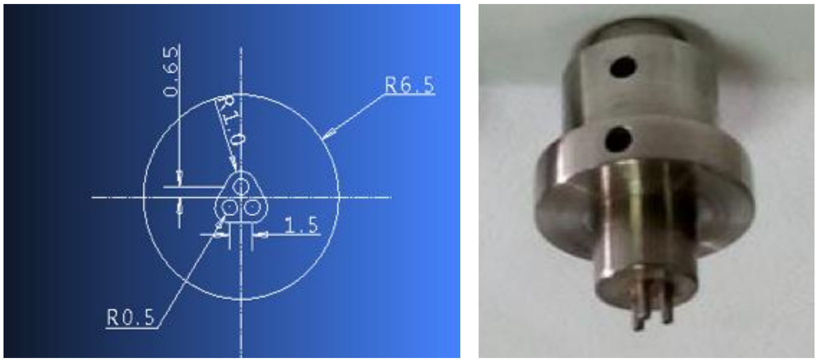

Fig. 1: Detailed design of triangular spinneret configuration.

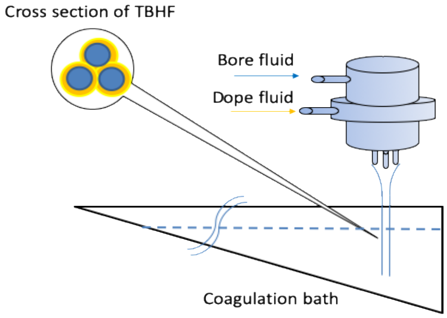

Fig. 2: Schematic diagram of spinning process using three holes spinneret.

During spinning, the dope and bore fluids were supplied at specified flow rates through the spinneret by two peristaltic feeding pumps. After entering the coagulation bath, the as-spun TBHF membranes were immersed in tap water for a few days to complete solvent removal, then, stored in a water bath with Glycerin to prevent the membrane cracks. Subsequently, the wet fibers were dried for 12 h, at the ambient atmosphere, the precipitated membranes were collected by a take-up roller. Each part of the spinning system is implemented and assembled as a preparation line. To avoid the uneven distribution of polymer dope, the bore fluid, and polymer dope was extruded from the top and the side of the spinneret, respectively. The detailed spinning operating conditions of TBHF membranes were summarized in Table (2).

Table 2: Spinning conditions of (MBHF) membranes.

Dope composition	PVDF/NMP/LiCL/EG:13/77/5/5
Bore fluid	Water
Dope flow rate, cm^3/s	1.14
Bore fluid flow rate, cm^3/s	1.1
Air gap distance, cm	0
Coagulation bath	Water
Temperature, ° C	25
Take-Up speed	Free fall

2.3 TBHF Module Fabrication

The TBHF module was prepared by loading the TBHF membranes into a module holder assembled from two connected PVC tube one-half inch with 3 and 12 cm length. Both ends of the module were sealed with epoxy. The module contained from five to seven TBHF with an adequate length of 14–15cm. The fabrication procedure consists of four steps: (1) shell preparation, (2) bundle preparation, (3) module assemble, and (4) epoxy casting.

2.3.1 Module Shell Preparation

The procedures for the preparation of a module shell are as follows:

a. Use a tube cutter to cut two pieces of one-half inch PVC tube with 3 and 12 cm length, and smooth the inner edge of cross-sections at two ends.

b. Fabricate the shell by applying threads, and assembling the tubes with tee connection, and be sure for sealing purposes.

c. The two ends of the designed module and the tee connection also will associate to opening enable to connect to the rubber hoses (Figure 3)

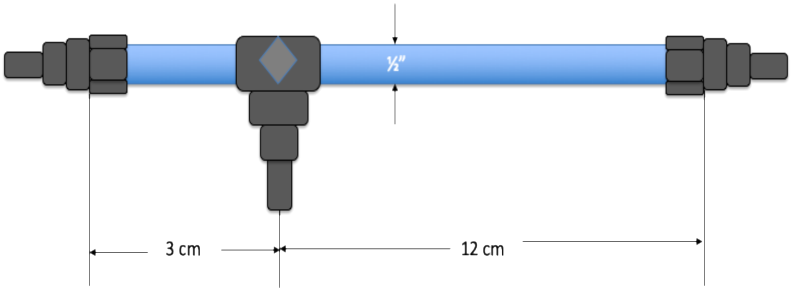

Fig. 3: A Multi Bore Hollow Fiber module casing

2.3.2 Preparation of the Bundle of Multi Bore Hollow Fibers

a. With an assumption of ~45% packing density, calculate required fiber numbers and fiber length, approximately 20 cm, select the best non-defective fibers.

b. Put the fibers in parallel order and put them together like a fiber bundle (Figure 4A).

c. Wrap a stretch Parafilm on one end of the fiber bundle (Figure 4B). Cut the wrapped end with a razor blade to yield a smooth cross-section (Figure 4C).

d. Encircle this end with a thin thread (Figure 4D, top portion).

2.3.3 *Module Assembly*

a. Place the shell vertically on a holder and leave enough space under the module shell so that it can accommodate the fiber bundle (Figure 4E).
b. Put a long thread through the shell lumen, and tie with the thin string that is roped upon the fiber bundle as shown (Figure 4E, top portion).
c. Pull the long thread gently upwards so that the fiber bundle is housed in the shell at a designated position. The untied portion of the fiber bundle (the part outside the module) should be suspended freely and hung loosely; thus, the fibers become ordered and packed naturally when being pulled into the shell.
d. Repeat the step (c) in Section 2 and wrap the other end of the fiber bundle with a piece of Parafilm. Each end should emerge out of the module shell with a length of 10 mm (Figure 4F)

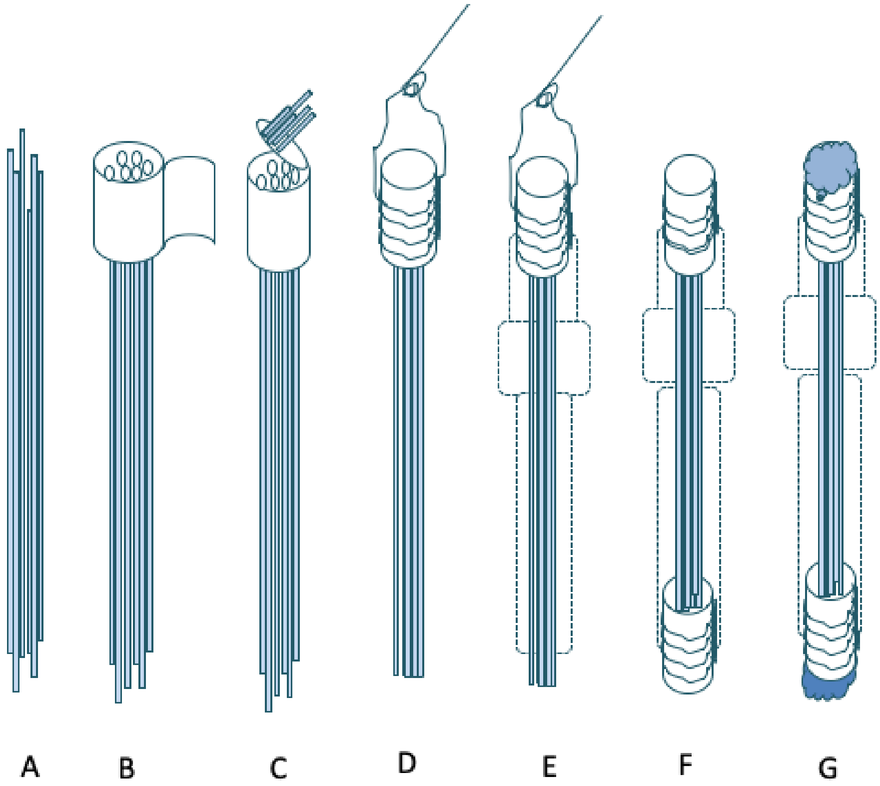

Fig. 4: Bundle preparation and module assembly

2.3.4 *Epoxy Resin Tube Sheets Casting*

Epoxy resins are usually employed to make tube sheets to isolate the permeate stream from the reject. The epoxy curing process is an exothermic reaction, and the heat generated speeds up the curing reaction that releases more heat. The heat may affect the membrane’s mechanical properties and morphology. The step-by-step procedure for the lab-scale module potting is given below.

a. Apply a layer of Araldite adhesive on the cross-sections of the bundle ends to seal each hollow fiber and prevent the creeping of epoxy through the fiber lumens by the capillary flow (Figure 4G).
b. Fabricate a disposable casting holder for the precise metering of epoxy flow during potting, as shown in Figure (5).
c. The bottom and side injection methods were used for potting epoxy as illustrated in figures (5A) and (5B), respectively.

d. Inside the injection method, fiber length was tall enough for keeping its ends outside the disposal casting holder and passing through a stretching parafilm sheet (Figure 6).
e. By applying a side injection method, the epoxy plugging problem, for the fiber lumen was solved.

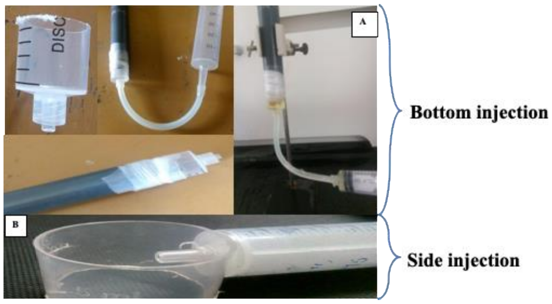

Fig. 5: Preparing for epoxy resin tube sheet casting.

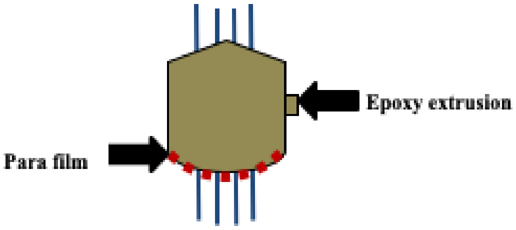

Fig. 6: Schematic diagram for epoxy side injection.

2.4 Membrane Characterization

Scanning electronic microscopy (SEM) was used to characterize the membrane morphology and surface topography. The dry samples were covered with a gold sputtering to provide for electrical conductivity. Pictures were taken on a JEOL 5410 scanning electron microscope (SEM) operating at 10 kV. The contact angle of the prepared PVDF membranes was determined by (SCA 20, OCA 15EC) using the sessile drop method (Preparation and finishing of cellulosic fibers, Textile research Division, NRC). The volume and contact time were 10µL and 10s respectively with five times for each measured membrane.

The membrane porosity is calculated by the percentage of pores volume to the total membrane volume.

$$\text{Porosity}\ \% = \left(\frac{\mathrm{V_p}}{\mathrm{V_m}}\right) * 100 \tag{1}$$

Where; V_p is the pores volume, and V_m is the volume of the membrane sample.

3 Vacuum Membrane Distillation (VMD) Experiment

The fabricated PVDF-TBHF membranes were evaluated using the VMD unit (Figure 7). The flat sheet and the TBHF membranes are tested using two modules; one of a diameter of 11.4 cm for the flat sheet membrane, and the developed module for the MBHF membrane. The feed for membrane testing is a NaCl solution. The operating conditions are the feed temperature of 65 °C and the volumetric feed flow rate of 8.6 cm3/sec. At the same time, a vacuum pump with a pressure of about -1 bar (absolute pressure 0 bar) connected to the permeate side of the membrane. The permeate collected and condensed, the permeate volume and salinity are measured. The water permeation flux (J_W) calculated according to the following equations:

$$J_W = \frac{V}{A} \tag{2}$$

Where J_W is the membrane flux; L/m^2.h, V is the volumetric flow rate of the permeate; L/h and A is the effective membrane area; m^2.

The salt rejection (SR) is obtained using the following expression, where C_P; the final salt concentration of the permeate stream andC_F; the initial salt concentration of the feed.

$$SR = \left(1 - \frac{C_P}{C_F}\right) * 100 \tag{3}$$

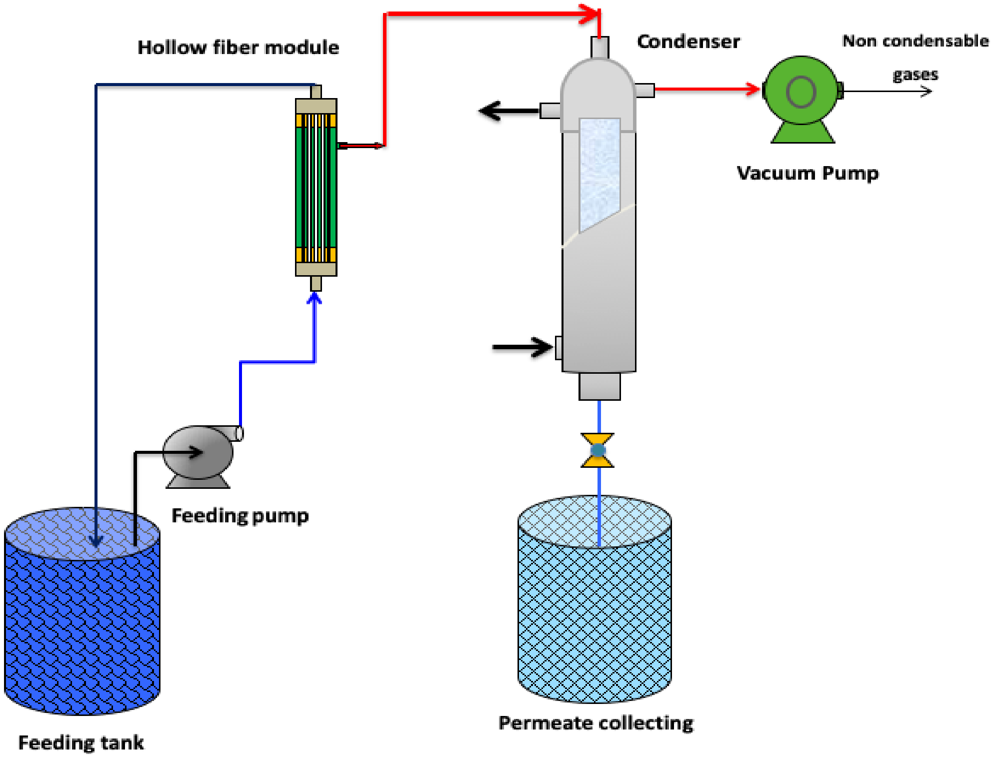

Fig. 7: Schematic diagram for VMD experiment.

4 Results and Discussions

4.1 TBHF Membrane Morphology

TBHF prepared from the blend of P1, which, gave the best results in VMD performance, and highest contact angle. Figure (8A) illustrates the Scanning Electron Microscopic graphs of the cross-section of P1 blends. It is clear that the blend P1 has well-distinguished porosity, and consisted of two layers; a spongy microporous layer that assists salt rejection, and the porous layer which facilitates mass transfer through the membrane. The TBHF membranes are prepared using the same blend of flat-sheet blend of PVDF (P1). An experiment was conducted at operating conditions as previously explained; the obtained TBHF fibers are characterized using Scanning Electron Microscope (SEM) to identify the fabricated membranes. The SEM of multi hollow channels of three bores with outer triangle geometry is represented in Figure (8B). The TBHF shapes have homogeneous consistent holes with an appropriate thickness.

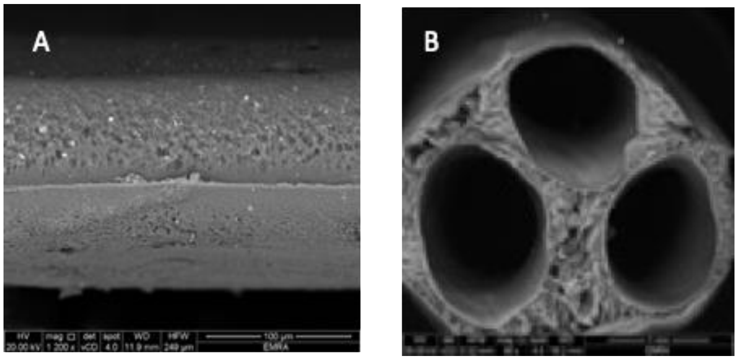

Fig. 8: Cross section morphology of the P1 membrane: A) flat sheet, B) TBF.

4.2 Contact Angle

The hydrophobicity of the prepared PVDF membranes was assessed by the determination of the Contact Angle (CA) using the sessile drop method (Preparation and finishing of cellulosic fibers, Textile research Division, NRC). As shown in Figure (9A, B and C), the prepared membrane from different blends of P1 (PVDF), gave the maximum value of CA (128.755°), when compared to the other dope solutions.

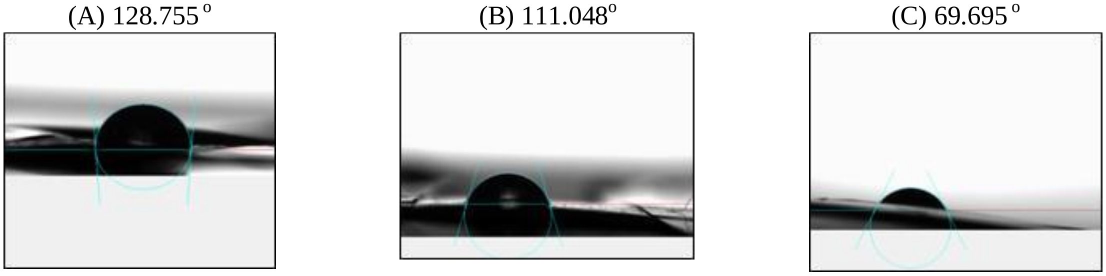
(A) 128.755 °
(B) 111.048°
(C) 69.695 °

Fig. 9: Water contact angles on A) P1, B) P1A (5%), C) P1T membranes.

The contact angle of the prepared Tri Bore Hollow Fiber (TBF) membrane (P1) is determined. Figure. (9A) illustrates the shape of the water drops on the prepared fiber (P1). The membrane contact angle is equal to 110° (Figure 10B).

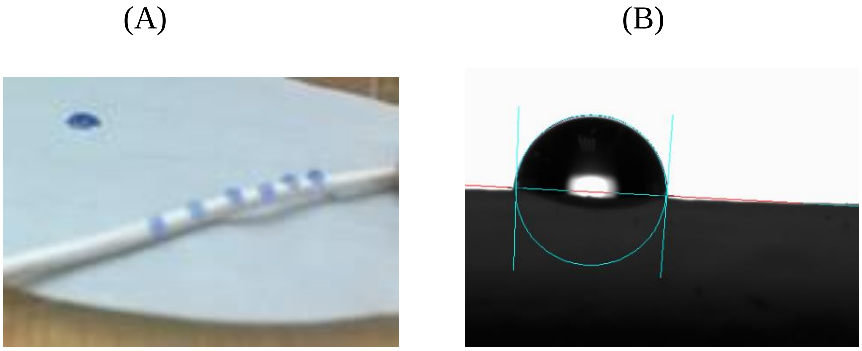
(A)
(B)

Fig. 10: Water drops appearance (A) and contact angle of P1 (B).

4.3 TBHF Module

The performance of TBF membranes was tested using the developed module (Figure 3). Figure (10) shows the steps which were followed to develop the multi bore hollow fiber module. The housing has two openings for the feed water and exit of the concentrate, respectively (Figure 11a). A bundle of triple-bore hollow fiber was wrapped with Para-film to facilitate its entering of the fiber bundle into the module as illustrated in Figure (11b), the bundle was drawn cautiously with a fine thread (Figure 11c) and enter it into the module when drawing it carefully (Figure 11d). These steps were repeated for the other side of the bundle (Figure 11e). The epoxy resin (purchased from the Modern Construction Chemicals, local market) was used to fill the spaces between the fibers. The epoxy forms a disc shape or tube sheet.

4.4 Evaluation of the Membrane Performance for VMD

4.4.1 Flat Sheet Membrane

Table (3) illustrates the performance indicators of the prepared membranes, in the form of the flux and salt rejection. It is clear from the Table (3) that the membrane flux is inversely proportional to the salt rejection. The maximum flux was 56.79 L/m^{2}h, while the salt rejection was 86%, and the flux is 22.12 L/m^{2}h, while the salt rejection was 99.3%, the former result is corresponding also to the maximum contact angle. Therefore, the selection of P1 (28.32 L/m^{2}h and 98.8%) is excellent and reliable.

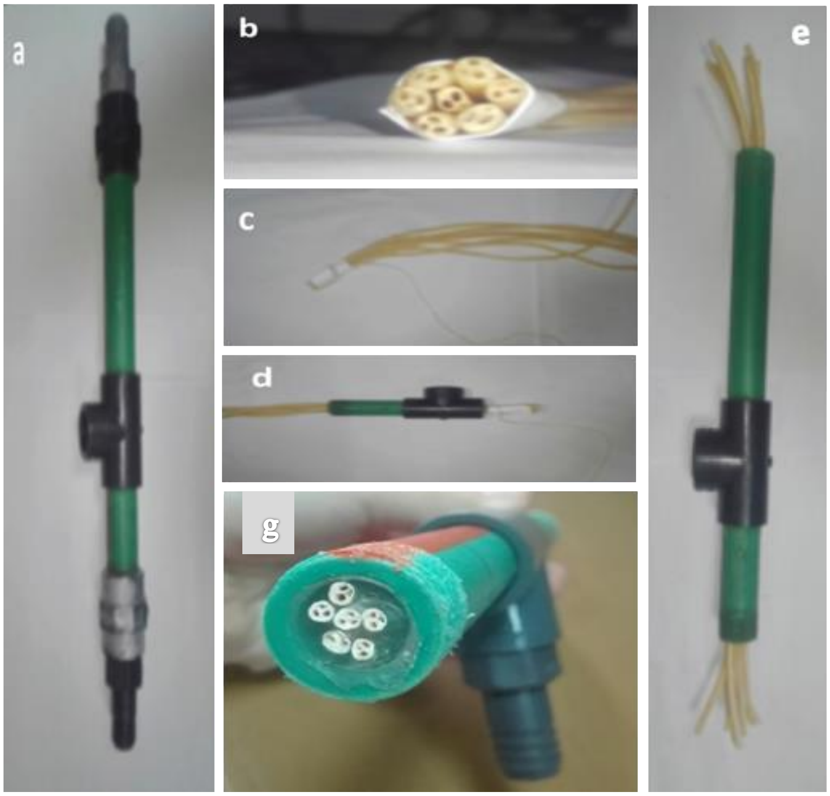

Fig. 11: Development of MBHF module.

Then, carefully cut the fibers at two ends to fix the caps with the module openings to obtain the final shape (Figure 11g).

Table 3: The flux and salt rejection of different membrane blends.

Blend	Thick, µm	Flow rate, cm^3/min	Time, min	Flux, L/m^2.hr	Rejection %
P1	200	230	30	40.71	84.33
P1	350	125	30	22.12	99.3
P1	**400**	**160**	**30**	**28.32**	**98.8**
P1A (5%)	200	190	20	50.44	70
P1A (5%)	2*200	230	30	40.71	84
P1A (5%)	**300**	**155**	**30**	**27.43**	**92.3**
P1A (2%)	300	246	23	56.79	86
P1T	200	105	20	27.88	83

4.4.2 TBHF Membrane

The fabricated PVDF TBF membrane evaluated using the VMD unit (Figure 12) using the developed module for the TBF membrane. The feed for membrane testing is a NaCl solution. The operating conditions are the feed temperature of 65 °C and the volumetric feed flow rate of 8.6 cm3/s. At the same time, a vacuum pump with a pressure of about -1 bar (absolute pressure 0 bar) was connected to the permeate side of the membrane. The permeate collected and condensed, the permeate volume and salinity are measured. The obtained flux was 44.3 L/m2.h, and the rejection was 97%.

Fig. 12: VMD system using to assess the performance of the TBF membrane.

5 Conclusion

The prepared Tri-Bore Hollow Fiber membrane is considered the second generation in the domain of membrane preparation. The testing and evaluation of such membranes are an essential issue, it proved, that it is an excellent choice for the application in the Membrane Distillation for water desalination. Although The development technique of the TBHF module still needs further development to be convenient for industrial applications, it is indicative and significant for further module scaling-up or industrial applications of membranes for membrane distillation or other membrane separations.

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