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Morphological Study of Fabricated PVDF Based Hydrophobic Membrane for Different Additives and Coagulation Bath Temperature

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Abstract: The demand of membrane distillation (MD) has increased since last few decades for numerous applications. The membrane used in MD is hydrophobic; therefore, the focus has been emphasised on the development of a suitable membrane with desired microstructure. In this study, the flat sheet hydrophobic membrane of suitable properties has been casted with various additives such as water, ethane-di-ol, and propan-2-ol in dope solution using a non-solvent induced phase separation (NIPS) technique. The effect of water content in dope solution has been studied on casted membrane porosity and contact angle. The maximum contact angle and porosity were found to be 96° and 53.23% at 4 weight percent of water content in dope solution of PVDF polymer and di-methyl-acetamide as solvent. It was found that SEM micrograph when ethane-di-ol and propan-2-ol are used as an additive shows more finger-like pores and nodules, respectively, in the microstructure of the casted membrane. Furthermore, synergistic effects using water with other additives were also identified using SEM micrograph of casted membrane and it was observed that water with ethane-di-ol and propan-2-ol form contact angle of 98° and 105°, respectively, for 2 weight percent each additive in dope. In this study, the membrane was also cast by dissolving PVDF powder in di-methyl-acetamide solvent with lithium chloride and the effect of the temperature difference between coagulation bath and film temperature was investigated using an SEM micrograph. Overall, it was found that water content and temperature difference aid in developing hydrophobic porous membrane of desired properties for MD applications.

Key words: PVDF, morphological study, contact angle, hydrophobic, non-solvent induced phase separation.

Introduction

The water availability for human use is declining rapidly due to the gradual rising of environmental pollution. Numerous processes are available for desalination such as reverse osmosis, adsorption, nanofiltration, and membrane distillation (Baghel et al., 2017; Singh et al., 2013; Upadhyaya et al., 2016a). The process of membrane distillation (MD) has certain benefits in comparison to other separation processes as it performs 100% rejections of ions and other non-volatiles (Baghel et al., 2018, 2020; Kalla et al., 2016), works at a lower pressure as compared to conventional pressure-driven membrane separation processes(Singh et al., 2017; Kalla et al., 2018, 2019), lower operating temperature and vapour pressure than conventional distillation, high water recovery (Geng et al., 2014; Kalla et al., 2019; Upadhyaya et al., 2016b, less energy consumption and greater selectivity.

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It is noteworthy, that the membrane used for MD application should be hydrophobic, high porosity, small thickness and interconnected open structure. Researchers (Pal et al., 2020; Shao et al., 2019) have fabricated these membranes using polymeric powder such as poly-vinylidene fluoride (PVDF), polypropylene (PP), polytetrafluoroethylene (PTFE), etc. Numerous techniques such as stretching, track etching, phase separation, electro spinning, etc., were adopted by different researchers (Feng et al., 2018; Tabatabaei et al., 2009) in order to prepare MD membrane for desalination. However, out of all these techniques, Kuo et al. (2008) found that the phase separation technique is more effective due to its simplicity and capability to fabricate membrane.

The key approaches adopted to improve membrane properties are membrane fabrication process parameters which include the polymeric concentration in dope, coagulation bath composition, film casting conditions, choice of solvent and non-solvent system (Guillen et al., 2011). In order to enhance some specific properties of the fabricated membranes for certain applications, some researchers (Deshmukh and Li, 1998; Hou et al., 2009) have also used various hydrophilic additives such as lithium chloride (LiCl), polyvinyl pyrrolidone (PVP), phosphoric acid (H_3PO_4), glycerol, polyethylene glycol (PEG) and H_2O, etc. It is noticed by the researchers that the demixing rate of solvent and non-solvent at the time of phase inversion is the key that helps in affecting the membrane morphology. Therefore, the choice of solvent and non-solvent system greatly affects the rate of demixing and becomes one of the deciding factors for membrane morphology. This scientific approach gives an idea for the pre-gelation step at the nucleation site therefore, Zheng et al. (2016) used water as a strong non-solvent additive in the dope and observed that it helps in the nucleation site for solidification and enhances the pre-gelation process at phase inversion due to moderate demixing.

Despite intensive research in membrane fabrication, the literature lacks an in depth understanding about the fabrication of hydrophobic membrane for better. Moreover, thermodynamic and kinetic aspects as discussed in the previous section are also not studied thoroughly in the literature. In this view point, the temperature difference between the coagulation bath and casting film may greatly affect the thermodynamic aspect of NIPS wherein control demixing rate between solvent and non-solvent and plays a vital role in deciding the membrane morphology during phase separation. In this work, the aforementioned gaps were taken into account for PVDF hydrophobic membrane fabrication suitable for MD applications and the effect of strong non-solvent as water concentration in dope solution on fabricated PVDF membrane properties such as porosity and contact angle were studied in terms of kinetic aspects. Moreover, the effect of additives such as ethane-di-ol and propan-2-ol with and without non-solvent additives as water in dope solution on the prepared flat sheet membrane morphology and their properties were also investigated for better understanding. Furthermore, in this work, the thermodynamic study of the temperature difference between coagulation bath and film casting solution is performed for simultaneous heat and mass transfer involved during phase separation from membrane morphology of casted PVDF membrane using lithium chloride and DMAc, as an additive and solvent, respectively.

Materials and Experimental Methods

In this section, chemicals procured and experimental procedures for membrane casting are described.

Materials

PVDF powder (FR-904) of specific gravity 1.77-1.79, was procured from Sita Chemicals Pvt., Ltd. (India) whereas di-methyl-acetamide (DMAc) of 99.5% AR grade with the density of 0.940 g/ml as a solvent was purchased from TECH INC India Pvt. Ltd. Ethane-di-ol, propan-2-ol, as an additive for dope solution was purchased from Savita Chemical Co., Ltd. (India) whereas lithium chloride (LiCl) of 99% AR grade was used as another additive and ethanol as non-solvent was procured from LOBA Chemicals. Non-woven fabric sheet (0.1 mm thick) was used as membrane support in order to avoid shrinkage during drying. The non-woven fabric was purchased from Permionics Membranes Pvt. Ltd, Vadodara, Gujarat.

Experimental Procedure

The membrane casting process comprises mainly two steps *viz.,* dope solution preparation followed by membrane casting. The detailed description of the experimental procedure is discussed in the subsequent section and the schematic diagram is represented in Figure 1.

Dope Solution Preparation

The dope solutions were prepared by dissolving the PVDF powder (16 weight %) in DMAc solvent along with additives either water, ethane-di-ol, propan-2-ol, or a mixture of water with the latter two chemicals. As per

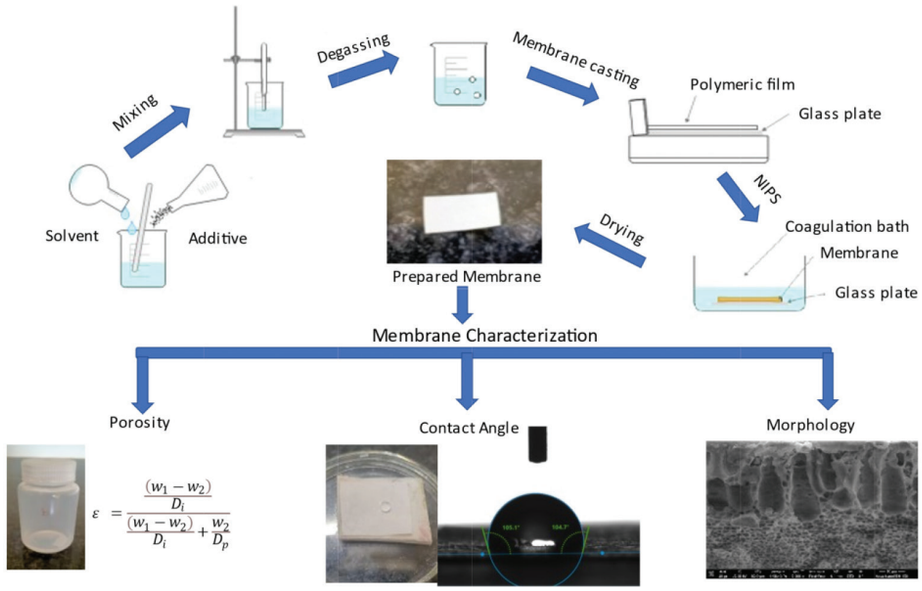


Figure 1: Flow Diagram for membrane preparation and characterisation.

composition specified in Table 1, the concentrations of H_2O as an additive in making dope solutions were kept under the range 1-4% whereas for making other dope solutions whose codes are represented in Table 1, the concentration of additives was taken the same as 4% by weight in making the individual membrane. Moreover, to study the synergistic effect of both additives with water, the composition of ethane-di-ol and water, as well as propan-2-ol with water, were taken 2% (each) by weight.

Table 1: Membrane code and its compositions for dope solution

*Membrane code**	*Additive used*	*Weight percentage of additive*	*Weight percentage of DMAc*
PVDF-H_2O-1	H_2O	1	83
PVDF-H_2O-2	H_2O	2	82
PVDF-H_2O-3	H_2O	3	81
PVDF-H_2O-4	H_2O	4	80
PVDF- ethane-di-ol	ethane-di-ol	4	80
PVDF- propan-2-ol	propan-2-ol	4	80
PVDF-ethane-di-ol + H_2O	ethane-di-ol + H_2O	2 + 2	80
PVDF-propan-2-ol + H_2O	propan-2-ol + H_2O	2 + 2	80

Once the recipe for each experimental run was prepared, the dope solution was stirred for 24 hours to ensure total dissolution of polymer and other chemicals on a temperature-controlled magnetic stirrer at 70˚C and 250 rpm. Subsequently, the solution was left overnight without agitation to allow for degassing at room temperature. After degassing, the viscosity of the dope solution was measured using a Labman (LMDV-60) rotating viscometer with Spindal-4 with 1.5 rpm spindle speed in which the degassed dope solution was poured into the cylindrical vessel and the spindle was submerged in the solution. Once the spindle began rotating, the viscosity of dope solution upon stabilisation was measured after 2 minutes in order to ensure the suitable range (1000-3000 mPa.s) of dope solution for membrane casting.

Membrane Casting Process

The dope solutions prepared were used to cast the membrane using NIPS. Firstly, a sheet of non-woven support fabric was cut in a particular dimension and fixed over a glass plate to provide a flat smooth surface for membrane casting. Thereafter, the glass plate was fixed on the flat sheet membrane casting machine. About 10 ml of dope solution was poured homogenously in the trapezoidal channel of the machine and cast over the non-woven fabric by pulling blade throughout the length of fabric using rotor and thickness was kept 200 microns. At room temperature, the casted membrane along with the glass plate was fully submerged into the coagulant bath containing 500 ml distilled water

as non-solvent for the phase separation process. The casted membrane was kept dipped in the bath for 24 hours. After phase separation, the membrane was dried at room temperature for 48 hours to ensure total removal of non-solvent from the membrane matrix and lastly, the casted membrane was used for characterisation.

Membrane Characterisation

The casted membranes were examined where the porosity was determined using the gravimetric method, while contact angle was calculated using the sessile drop method. The membrane morphology was identified using scanning electron microscopy (SEM).

Porosity

The gravimetric method (Abdel-karim et al., 2018) was used to measure the membrane porosity. Each casted membrane was cut into a rectangular strip of dimension 1×2 cm and then immersed in propanol for 24 hours at room temperature so that the solvent could penetrate completely inside the pores of the membrane. After weighing, the samples were gently dabbed with tissue paper to remove the excess propanol and then weighted again. Porosity was estimated by using equation

$$1\varepsilon = \frac{\frac{(w_1 - w_2)}{D_i}}{\frac{(w_1 - w_2)}{D_i} + \frac{w_2}{D_p}} 1$$

where w_1, w_2, D_i and D_p are the weight of the wet membrane in g, weight of dry membrane in g, 2-propanol density in g/m^3, and polymeric membrane density in g/m^3, respectively.

Hydrophobicity Determination using Contact Angle

The contact angle of the water droplet over the casted membrane was measured using an instrument Drop Shape Analyzer (DSA25) Mk2 of firm Kruss, Germany. A water droplet of 4µl was placed over the flat surface of the membrane and digital image was captured as shown in Figure 2, using Kruss advanced drop shape v2.4.7 to determine the geometrical angle between the hydrophobic surface and water droplet.

Morphology

A scanning electron microscope (SEM) was employed to observe the cross-sectional view of the membranes in order to understand the overall microstructure of the membrane. Membranes were cut to a square coupon and frozen in liquid nitrogen, then broken, which were deposited on the copper holder and their

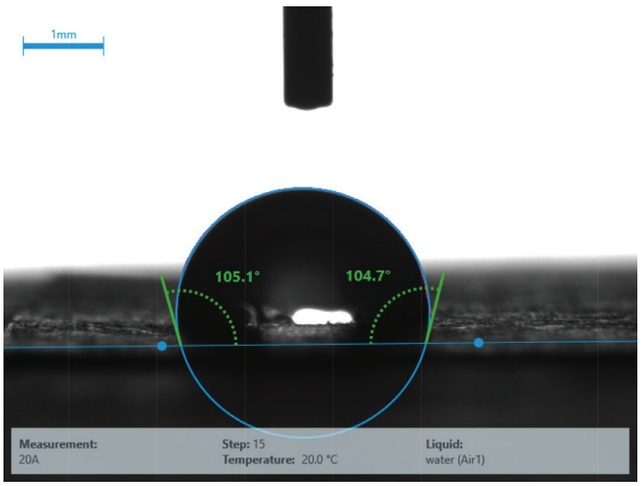


Figure 2: Image of the sessile drop to measure contact angle.

cross-sectional surface was sputtered with gold to make it conductive. Their SEM images were captured at different magnifications to examine the effect of different additives on morphology.

Results and Discussions

Effect of Strong Non-solvent (H_2O) Additive on Porosity and Contact Angle

The effect of strong non-solvent water on percentage porosity and contact angle is illustrated in Figure 3. It was observed that on increasing the weight percent of water from 1 % to 4% in dope solution, the porosity and contact angle of the casted membrane increased from 44.12% to 53.23% and 86° to 96°, respectively. This increase in porosity may be explained by the path of composition change in the ternary phase diagram of polymer/solvent/non-solvent system as shown in Figure 4. Path 1 shows the spinodal decomposition in which the composition change passes through the critical point, whereas path 2 shows nucleation and growth in which the solution passes slowly through the metastable region (Zhao et al., 2013). On increasing the water content in dope solution, rapid demixing takes place during coagulation. Water is a strong non-solvent, so when added in dope solution it promotes partial phase inversion before the coagulation process starts, which provides enough time for solid-liquid demixing which results in high porosity and interconnected pores. The pre-gelation process shows a strong trend when H_2O was added because H_2O may even act as the nucleus for the solidification process, as a result porosity will get increased.

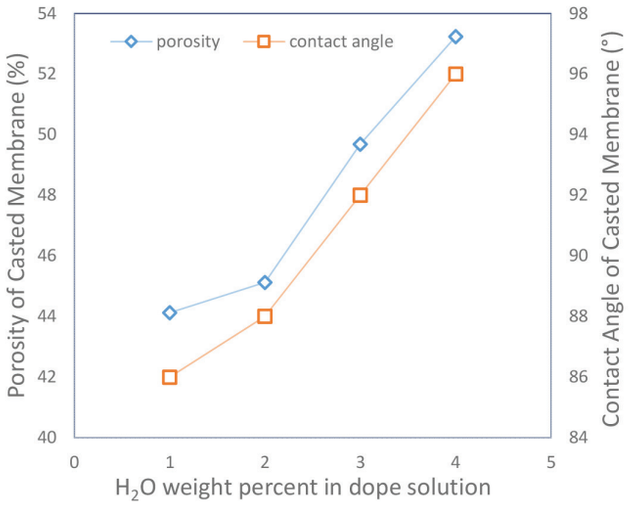


Figure 3: Effect of water content on porosity and contact angle.

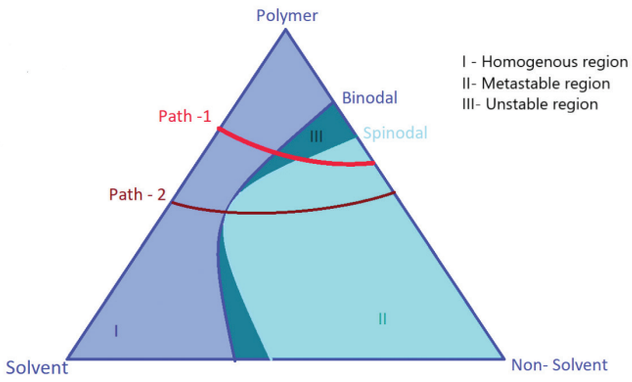


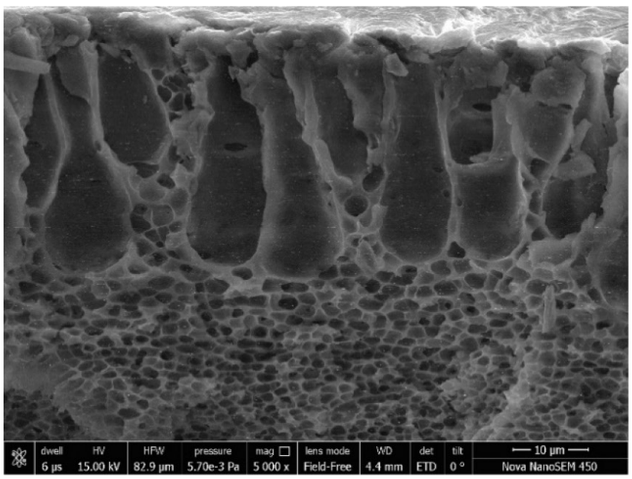
Figure 4: Ternary phase diagram between solvent, non-solvent and polymer.

The reason for the increment in contact angle with the weight percent of water in dope may be attributed to the fact that the time required for solid-liquid demixing gets increased. As a result, a thin polymeric layer makes the membrane asymmetrical. This pre-gelation creates a high concentration of PVDF polymer on the top layer. Another fact may also be understood from Young's expression.

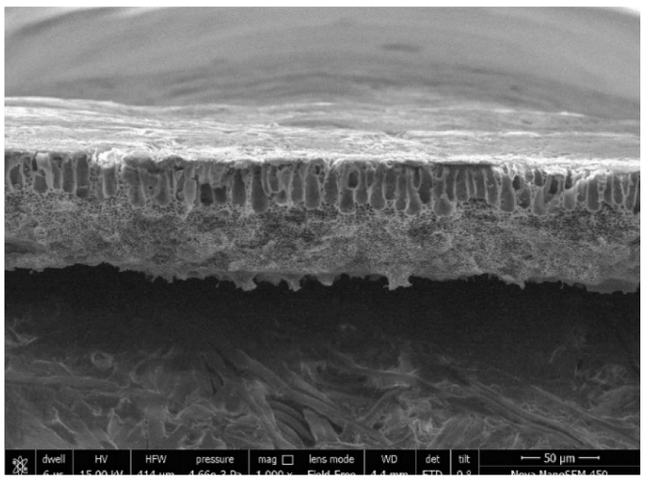
Effect of Ethane-di-ol and Propan-2-ol in Dope Solution on Morphology

As per the composition mentioned in Table 1, the cross-sectional SEM image of casted flat sheet membrane using ethane-di-ol (4 weight %) in dope solution is represented in Figure 5(a,b) at different magnifications of 5000× and 1000×, respectively. It is evident from both micrographs that the microstructure seems spongy and integrated with some globules and the membrane skin looks more porous with an inhomogeneous pore size as a result of asymmetric structure. From the image, it is also revealed that this asymmetrical structure has a porous skin layer on the top followed by a finger-like structure and underneath a sponge-like structure. The long and deeper pores on the top side of the membrane may be due to delayed demixing of the solvent and non-solvent in NIPS.

Similarly, the casted flat sheet membrane cross-section view is depicted in Figure 6(a,b), prepared using propan-2-ol (4 weight %) in dope solution. From the micrograph, it is anticipated that symmetric membrane structures are composed of two layers of macro-voids with uniform nodules. This phenomenon could be attributed to the occurrence of gelation induced by



(a) Ethanediol 4 wt% (5000 magnification).



(b) Ethanediol 4 wt% (1000 magnification).

Figure 5: Cross sectional micrograph of casted PVDF membrane with ethane-di-ol.

crystallisation before liquid-liquid demixing with the formation of nodules, which when dipped in coagulant get converted into macro voids. The rate and extent of phase demixing determine the overall membrane structure and mechanical properties. It is also observed that the SEM image shows some big nodules on the upper cross-section side of the membrane.

Synergistic Effect of Non-solvent H_2O along with Ethane-di-ol and Propan-2-ol

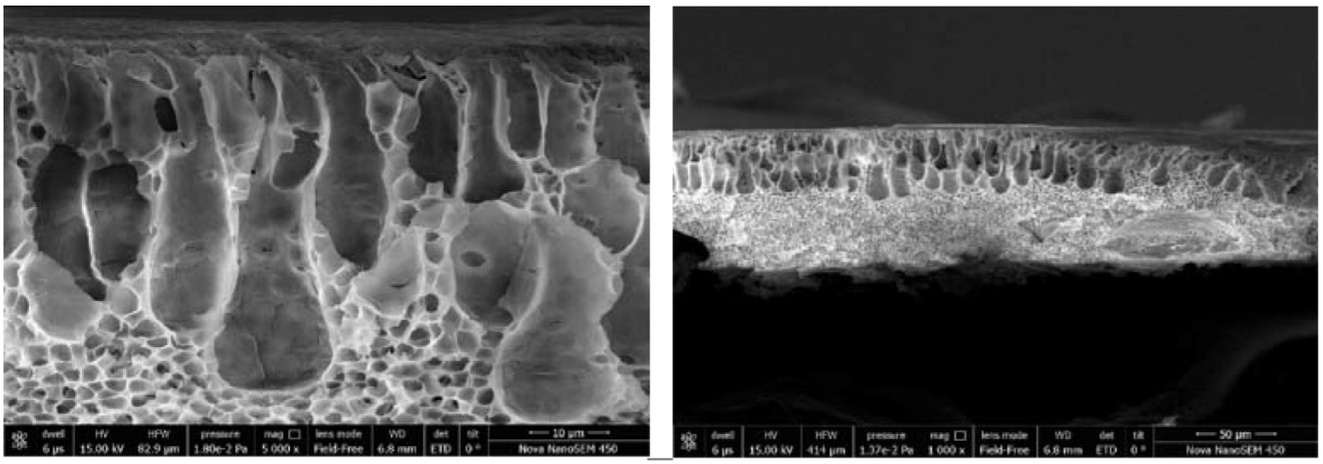
The recipe compositions of the additives were taken as per Table 1 and cast membrane porosity, as well as contact angle, is shown in Table 2. It is observed from Table 2, that water along with additives, namely ethane-di-ol and propan-2-ol, enhances the porosity and contact angle of the membrane as compared to using individual additives. This synergistic improvement in porosity and contact angle may be because water increases the interconnectivity of the pores in the membrane matrix, which can be understood while comparing Figure 7 with Figure 5 and Figure 8 with Figure 6. More elaborately, by the addition of water in the dope solution, the partial phase inversion takes place before the coagulation process. This provides enough time for solid-liquid demixing to form high porosity and interconnecting pores; as a result, giving rise to anasymmetric membrane. In addition, due to the thin polymeric film over the membrane, the contact angle of the membrane also gets increased. The enhanced interconnected structure reduces the tortuosity of the membrane that can be visualised in Figures 7 and 8, which in turn may increase the permeate flux. This interconnected structure is formed because of the low speed of the liquid-liquid demixing rate at the time of coagulation.

Table 2: Porosity and contact angle of PVDF membrane with additives

Additive	*Porosity (ε)*	*Contact angle (°)*
Ethane-di-ol (4 wt %)	49.793	87
Propan-2-ol (4 wt%)	46.423	94
Ethane-di-ol (2 wt%) + H_2O (2 wt%)	50.7235	98
Propan-2ol (2 wt%) + H_2O (2wt%)	48.957	105

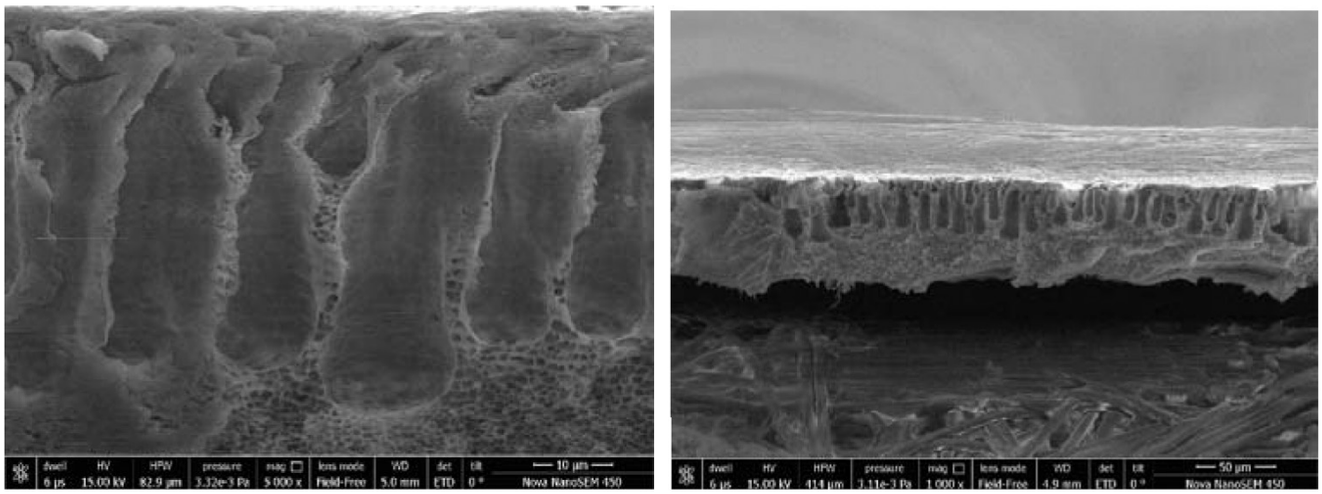
Effect of the Temperature Difference between Coagulation Bath and Casting Film

In this study, the PVDF membrane was prepared using LiCl (4%) and DMAc (80%) as an additive and solvent respectively, by weight and the effect of temperature difference (30 °C and 0 °C) at the time of film casting and coagulation bath temperature on membrane morphology is investigated. The cross-sectional morphology of casted membrane is illustrated in SEM micrographs shown in Figure 9(a,c) at 2500×, and 1000× magnifications, respectively for a temperature difference of 30°C between film casting and coagulation bath. It is observed from the figure that long, deep, and wide pores are formed throughout the membrane. The reason for this kind of morphology may be the large temperature difference between film dope solution and coagulation bath, which enhances the demixing rate of solvent and non-solvent. However, when the membrane was cast by maintaining the same temperature of coagulant and film,



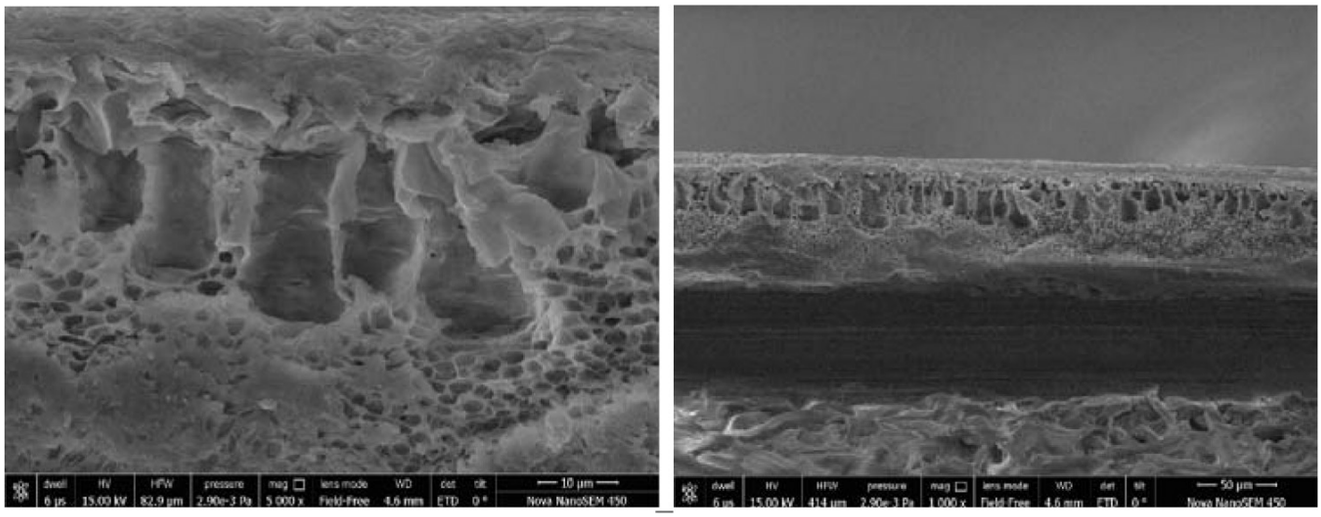
(a) Propan-2-ol 4 wt% (5000 magnification). (b) Propan-2-ol 4 wt% (1000 magnification)

Figure 6: Cross sectional micrograph of casted PVDF membrane with propan-2-ol.



(a) Ethanediol + H_2O (5000 magnification). (b) Ethanediol + H_2O (1000 magnification).

Figure 7: Crosssectional micrograph of casted PVDF membrane with H_2O + Ethane-2-ol.



(a) Propan-2-ol + H_2O (5000 magnification) (b) Propan-2-ol + H_2O (1000 magnification).

Figure 8: Crosssectional micrograph of casted PVDF membrane with H_2O+Propan-2-ol.

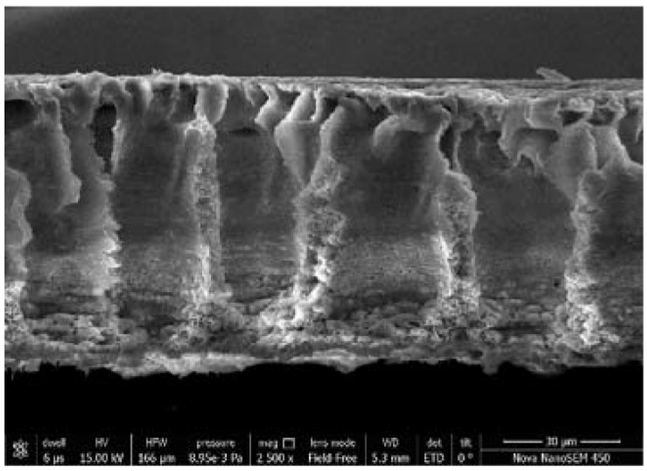
the asymmetrical structure of membrane was observed as represented in Figure 9 (b, d).

It can be easily visualised from the micrographs that finger-like pores at the upper layer of the membrane and sponge-like pores at the bottom with a thin polymeric layer at the top are formed. This typical structure of membrane can be corroborated due to zero heat transfer rate, which, in turn, slows the mass transfer rate as the migration now takes place due to concentration difference but not by temperature difference. Henceforth, gradual liquid-liquid demixing will take place.

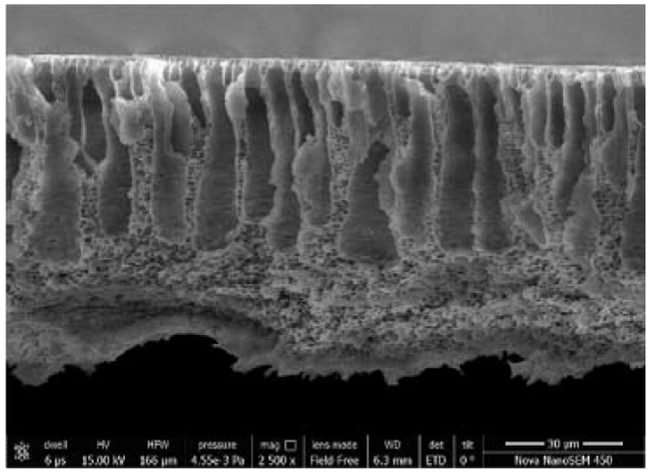
As a result, it can be understood that by adjusting the temperature of the coagulation bath and film casting dope solution, the membrane of desired morphology with controlled microstructure can be prepared.

Conclusions

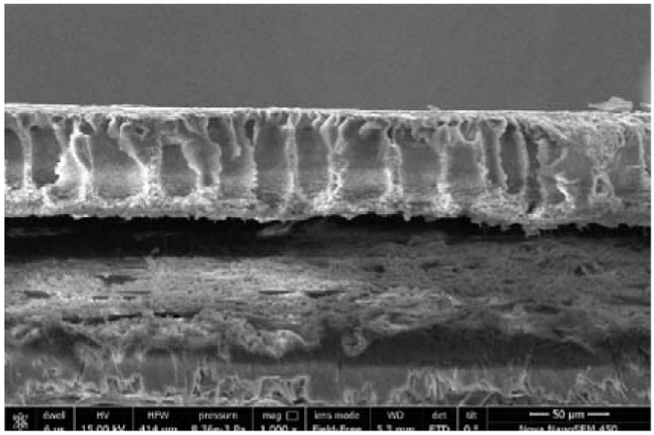
PVDF hydrophobic membrane was successfully prepared with DMAc as solvent and water (H_2O), ethane-di-ol, and propan-2-ol as additives in the dope by NIPS. It was found that water in the dope solution acts as a pore-forming additive. It was observed that ethane-di-ol and propan-2-ol enhance the porosity and contact angle of the casted membrane. However, the maximum porosity and contact angle were found to be 53.23%, and 96°, respectively, for water as an



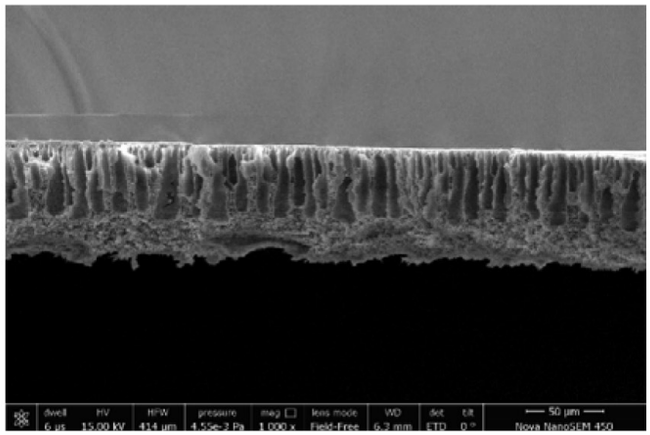
(a) Micrograph at 30˚C (2500 magnification).



(b) Micrograph at 0˚C (2500 magnification).



(c) Micrograph at 30˚C (1000 magnification).



(d) Micrograph at 0˚C (1000 magnification).

Figure 9: Effect of temperature difference between coagulant and film temperature.

additive as compared to 49.79% and 87° in case of using ethane-di-ol, whereas 46.42% and 94° in the case of propan-2-for 4 % of individual additive (by weight) in dope solution in different runs. Synergistic effects of water along with ethane-di-ol and propan-2-ol were also investigated and it was found that using water with these additives improves the porosity to some extent whereas the contact angle increased from 87° to 98° for ethane-di-ol and H_2O +ethane-di-ol, respectively, and 94° to 105° for propan-2-ol and H_2O + propan-2-ol, respectively. Furthermore, the effect of a temperature difference as driving force between coagulation bath and film casting solution was investigated. It was observed that on maintaining higher temperature difference, mass transfer rate and demixing rate get enhanced, resulting in macro size pores whereas, long uniform finger-like pores were formed on keeping the same temperature of bath and film. Therefore, it is obvious that water content and temperature differences play a vital role in developing suitable membranes.

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