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EFFECT OF QUENCHING TEMPERATURES ON THE PROPERTIES AND PERFORMANCE OF KEVLAR SUPPORTED IPP MEMBRANES

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ABSTRACT

Novel membranes were fabricated using weave Kevlar fabric sandwiched between two isotactic polypropylene layers and employing varying quenching temperatures. Thermally induced phase separation (TIPS) dip coating method was adopted to generate the micro-porosity in the composite membranes. SEM and AFM were used to directly observe and confirm the morphologies and micro-pores in the fabricated membranes. From SEM analysis, it is observed that with the increase in the quenching temperature for the fabricated membranes, relative porosity has increased. Average Pore sizes are observed to be 0.925 µm, 2.850 µm, and 3.250µm, respectively for 20, 30 and 40 ℃ quenching temperatures. The flux characteristics of the prepared membranes were also tested at various pressures using water, methanol, ethanol and isopropanol as solvents in a custom-made filtration cell. The obtained results indicate the dependence of flux on the type of solvent, pressure and membrane. The flux for the solvents observed to increase the membranes prepared at high quenching temperature and is attributed to the increase in the porosity.

Keywords: Thermally Induced Phase Separation (TIPS), Quenching Temperature, Membrane, Microporosity, Polypropylene.

I. INTRODUCTION

Porous membranes are the most simply understandable part of the membrane family. These membranes compose of a solid matrix with defined pores having diameters ranging from less than 1 nm to more than 10 µm[1-3]. Sieving mechanism are involved to separate different componentsinclude in the feed with the pore diameters and the particle sizes being the determining parameters. Various materials such as ceramics, graphite, metal or metal oxides, and many polymers are used to fabricate porous membranes. Their structure may be symmetric, i.e. the pore diameters do not vary over the membrane cross- section, or they can be asymmetric, i.e. the pore diameters increase from one side of the membrane to the other typically by a factor of 10 to 1000. The preparation techniquesof these membranes areirradiation, sinter processes, phase inversion,etching techniques and polymer precipitation procedures[4-6]. Porous membranes are used in microfiltration and ultra-filtration processes. They can be categorized according to their different characteristics such as (1) type of material, i.e. ceramic or polymer, hydrophilic or hydrophobic, (2) structure, i.e. symmetric or asymmetric, or (3) pore size and pore size distribution. The pore size is used to make a distinction between porous membranes. Microfiltration membranes normally have pore diameters larger than 20 nm, while ultra-filtration membranes have typical pore diameters between 2 nm and 20 nm[7-10]. Thermally induced phase separation (TIPS) is a well known technique to prepare polymeric micro-filtration membranes. Polypropylene (PP), polytetraflouroethylene, polysulfone, polyethylene, and polyvinyldene fluoride micro-porous membranes can be made using TIPS. In TIPS, the polymer is melted in a diluent having high boiling point such as dioctyle phthalate, bibutyle phthalate, soybean oil, etc. The homogenized solution is casted on a preheated glass/steel plate. The diluent is extracted from the casted film using appropriate solvent that generates porous structure in the virgin polymer. By varying the polymer, the polymer concentration, the diluent, and the casting plate temperature, micro-porous phase inversion membranes can be made with a very large variety of pore sizes, from less than 0.1 to more than 20 µm, and with varying chemical, thermal and mechanical properties[4, 11, 12]. The basic principle of microfiltration is portrayed in Fig. 1. The membrane that separates a feed solution from a filtrate has a symmetric porous structure with an average pore size between 0.1 to 10 µm.The driving force is a pressure gradient and if the concentration of components in the two phases separated by the membrane is not identical there will also be a chemical potential gradient acting as driving force for the mass transport across the membrane[13, 14]. However, mass transport in microfiltration membranes takes place exclusively through the pores. The solid membrane matrix can generally be considered as completely impermeable. Both solvents and solutes permeate the membrane by viscous flow through the pores. There may also be diffusion in the moving pore liquid if concentration gradients are established between the feed and filtrate solutions[15, 16].

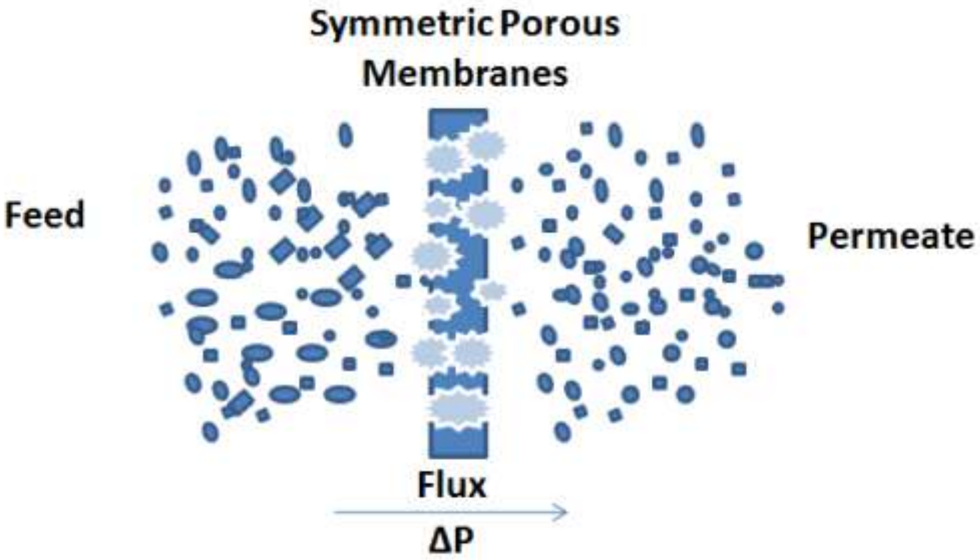

Figure 1. Schematic drawing illustrating the principle of microfiltration

In the present investigation, novel Kevlar supported isostatic polypropylene (IPP) micro-porous membranes are fabricated using dip coating allied with TIPS process. The effect of quenching temperatures on the pore size/density, and permeation of variant solvents through the fabricated IPP supported liquid membranes (SLMs) is discussed herein.

II. RESULTS AND DISCUSSION

The membranes prepared at different quenching temperatures are analyzed by SEM and AFM and their flux characteristics are measured for water, methanol, ethanol and iso-propanol. The results of these studies are discussed below.

2.1 SEM Analysis

(Fig.2-4) represent the SEM images of the IPP-membranes prepared by applying the quenching temperature of 20°C, 30°C and 40°C, respectively.

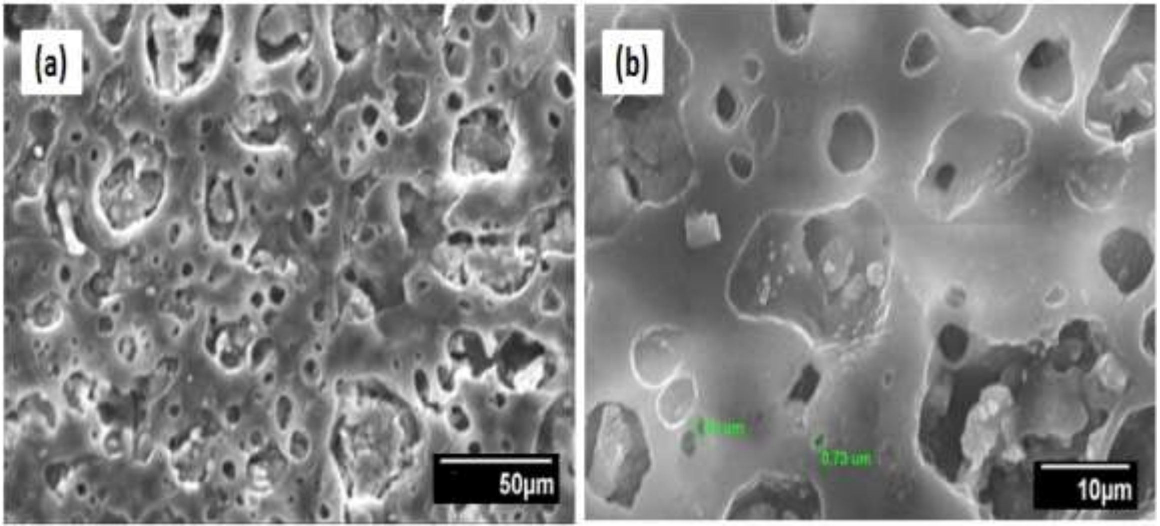

Figure 2. SEM images of the membrane prepared at quenching temperature 20°C

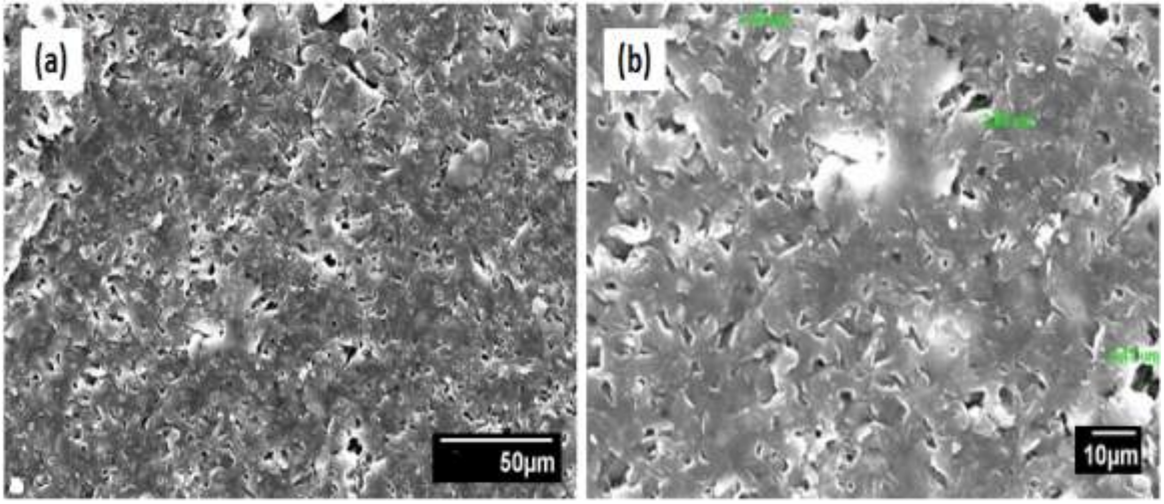

Figure 3. SEM images of the membrane prepared at quenching temperature 30°C

The effect of quench temperature on pore morphology was observed in SEM pictures at various magnifications to observe the cellular pores near the skin layer and dendritic pores on the non-skin layer. Quench temperature affected the pore size distribution remarkably. Pore sizes of resultant membranes were about 3.25 µm, and membranes that were prepared at lower quench temperatures had relatively lower porosities. It is evident from figures that the degree of quenching temperatures has significantly affected the pore morphology of the IPP-membranes. It is noted that with the increase of the quenching temperature, porosity of the prepared membranes has also increased[7, 11, 17].

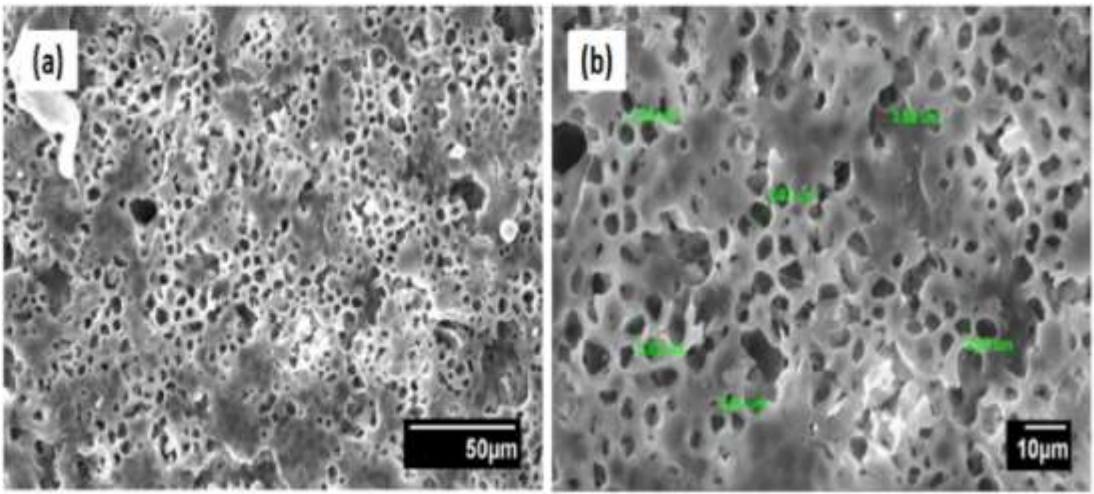

Figure 4. SEM images of the membrane prepared at quenching temperature 40°C

This observation is in good agreement with previously published results. Since, the quench temperature affects the phase separation mechanism during the process of membrane preparation, therefore, it is expected that the quench rate was slower when quench temperature was higher. This can cause the IPP crystallization to become slower during slow quenching, and the diluent become richer in the solid sample. This process thus results in the relatively higher porosity and larger pore size in the fabricated membranes, which were prepared at relatively higher quench temperature[18]. The cooling rate of the solution quenched at higher temperature has slow compared to the membrane fabricated at correspondingly lower temperatures. By reducing the cooling rate, pore size increases because of the larger time is available for coarsening of the droplets generated by the liquid-liquid phase separation[2,19-21].

2.2 AFM Study

Representative AFM images of the IPP-membranes prepared at the quenching temperature of 20, 30, and 40^{o}C is shown in (Fig. 5-7). The results of the AFM imaging show the details of the surface of the membranes and presence of pores on the surface topography of the prepared IPP-membranes. As supported by SEM analyses, presence of pores is thus also confirmed by AFM in the fabricated membranes[2, 22,23].

2.3 Flux Rate Studies

To see the performance and efficiency of the prepared membranes, the flux rate analyses were performed. For this purpose, four different liquids were used, which possess different structures, polarity and sizes. These liquids were:

- Water
- Methanol
- Ethanol
- Iso-Propaonol

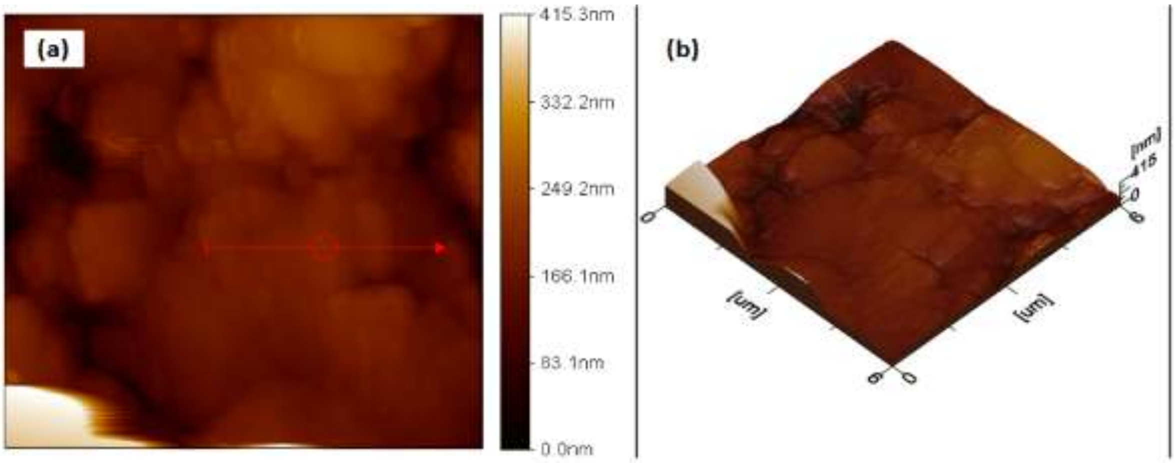

Figure 5. AFM topographical images of the membrane prepared at 20°C quenching temperature

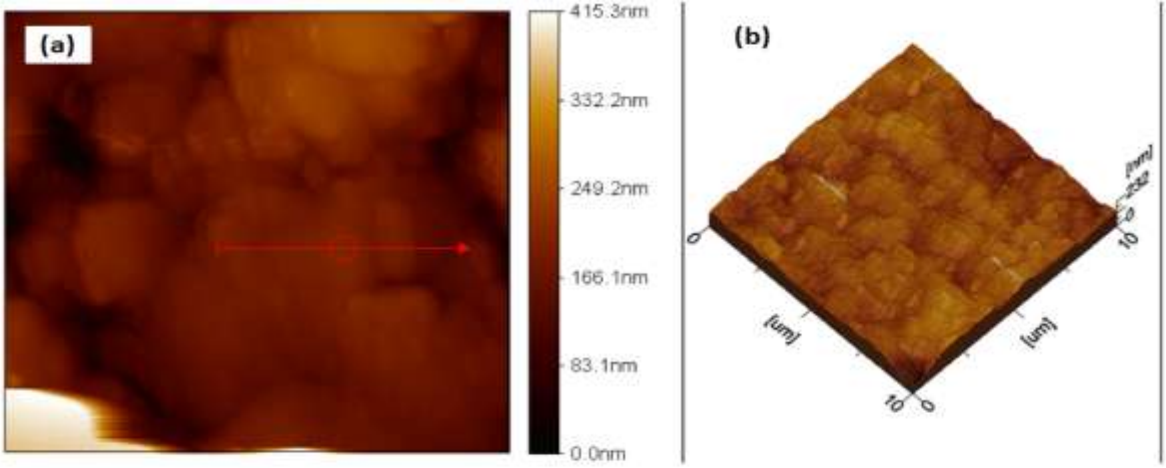

Figure 6.AFM topographical images of the membrane prepared at 30°C quenching temperature

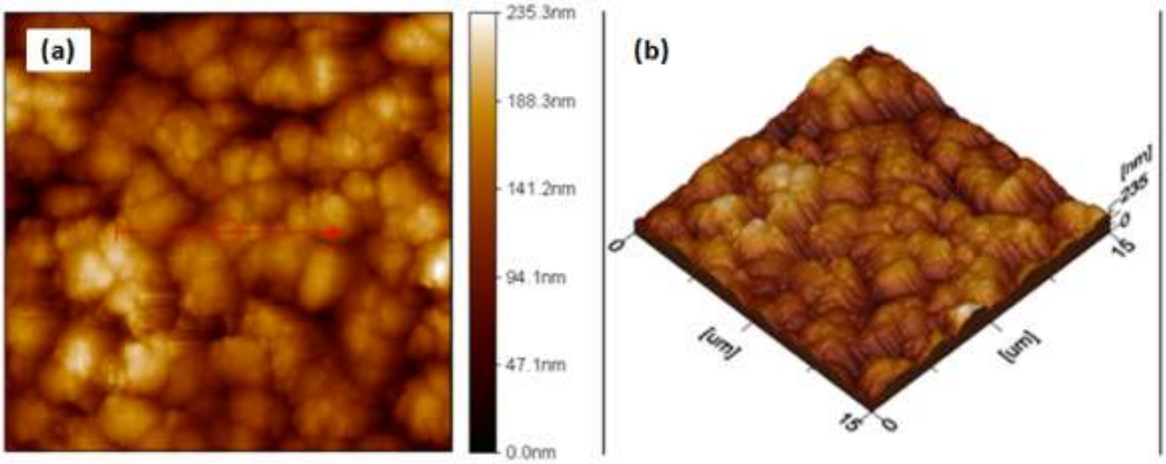

Figure 7. AFM topographical images of the membrane prepared at 40°C quenching temperature

Tabel1. Summary of properties of the liquids employed to test the performance of the prepared IPP Kevlar supported membranes.

Property	CAS #	Molar mass (g/mol)	Size (nm)	Density g/cm3 (20 °C)	Dipole moment (D)	Viscosity Pa*s (20 °C)
Water	7732-18-5	18.02	0.26	1.0	1.85	0.001
Methanol	67-56-1	32.04	0.41	0.7918	1.69	0.00059
Ethanol	64-17-5	46.07	0.44	0.789	1.69	0.0012
Iso-propanol	67-63-0	60.11	0.48	0.786	1.66	0.00286

In accordance to the data given in Table, size of the solvent molecules can be categorized as:Water<Methanol<Ethanol<Iso-Proponol Thus, Water has the smallest and isopropanol has the biggest molecular size among these solvents. In addition, as data given in Table indicate that these solvents are also different in terms of other characteristics such as density, dipole moment and viscosity. The correlation of all these parameters with the performance of the prepared membranes will be the subject of future work [24, 25]. In the current work, main focus is to interpret the data in accordance to the size of the solvent molecules as this provides information about the extent of porosity in the prepared membranes. During the course of the experimental work, N_2 pressures of 13 psi, 26 psi and 39 psi were applied and graphs between pressure and the flux rate were recorded[11,19, 26,27]. It is observed that with the increase in pressure, the flux rate of all solvent increased as expected. Furthermore, it is noted that in general water has the maximum flux rate and isopropanol has the minimum. This observation can be correlated to the size of the liquid droplets employed in the present work. (Fig. 8-11) represents the result of the flux rate of different solvents tested for the IPP-membranes prepared at various quenching temperatures of 20°C, 30°C and 40°C, respectively.The results obtained indicate the dependence of flux on the type of solvent, pressure as well as the membrane[28]. It is interesting to observe that the flux for the solvent increase with the increase of the quenching temperatures in the prepared membranes and thus can be attributed to the increase in the porosity in the prepared IPP-membranes[20-22]. As discussed earlier that the quenching temperature may have affected the degree of nucleation and hence crystallization, which can significantly modify the crystallization behavior of IPP.

It is possible that there is reduction in formation of crystalline, spherulites dimensions and thus reduction in the crystallinity. This effect may have resulted in relatively larger pores and also higher pore density to affect the porosity of the fabricated membranes[29-31]. The observed results are in good agreement with that of SEM for the IPP-membranes investigated in the current work**.** The flux-pressure curves illustrates three annotations; the first observation is that with increasing the gas pressure permeation of the corresponding solvent increases due to the enhancement in work per unit volume of or tendency of force to speed up the liquid permeation rate through the membrane; the second inspection is regarding the type of solvent, that for low molecule size solvent has the higher flux rate than the other ones owing to the ease to flow through the pores of the membrane and usually the permeation through the microfiltration membranes is mainly attributed through their pores[32].

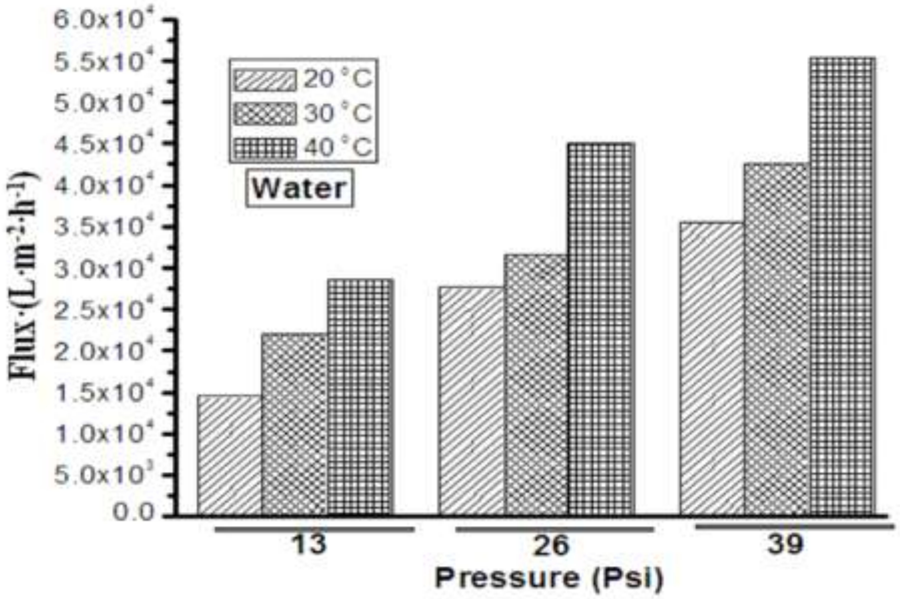

Figure 8. Water flux through the membranes prepared at variant quenching temperatures

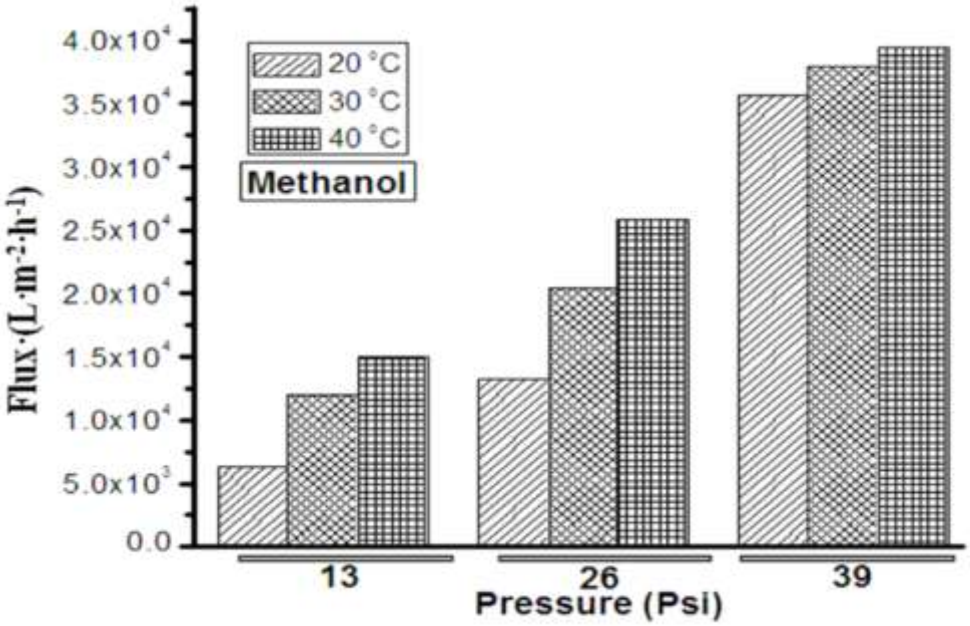

Figure 9. Methanol flux through the membranes prepared at variant quenching temperatures

The third study of this investigation is that with increasing the quenching temperature of the fabricated membraneduring its synthesis, solvent flux through the membrane enhances due to the pore size/density enhancement as observed in SEM images[1, 7, 9, 33].

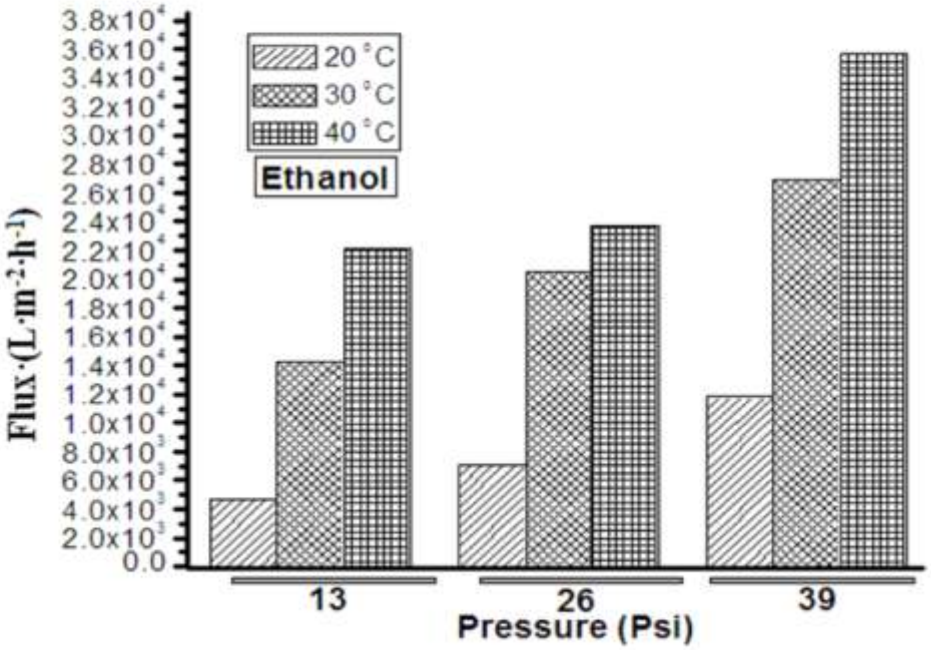

Figure 10. Ethanol flux through the membranes prepared at variant quenching temperatures

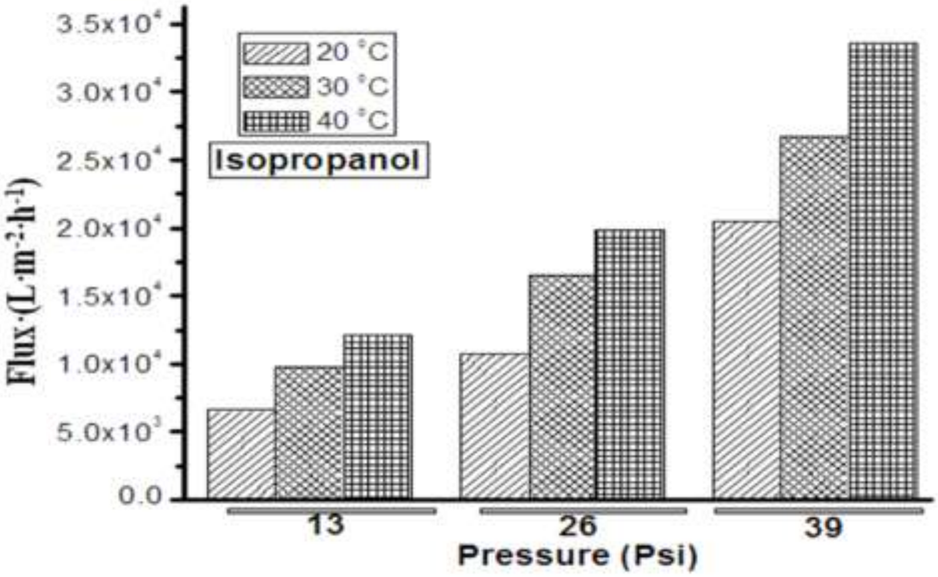

Figure 11. Iso-propanol flux through the membranes prepared at variant quenching temperatures

III. EXPERIMENTAL

3.1 Materials

The reagents employed to fabricate membrane were used as received. The membranes were fabricated from isotactic polypropylene (IPP) was supplied by wuhanxuyadi chemicals Co., Ltd. The melt flow index (MFI) value of the IPP was measured and found to be 14.2g /10 min. Adipic acid was supplied by sigma-aldrich, Germany. Soybean oil (diluents) was supplied by zhejiangjiaaoenprotech stock Co., Ltd. To support the membrane, Woven Kevlar Fabric (WKF) (DuPont International) was used. N- Hexane was supplied by Merck.

3.2 Fabrication of Membranes

The membranes were fabricated using thermally induced phase separation (TIPS) procedure and dip coating method. Typical example of the procedure is described below. In this technique, the materials which are immiscible at room temperature are subjected to a sufficient high temperature and thus become completely miscible. Typically, in the first step, required amount of soybean oil, IPP and nucleating agent was taken in a 500 ml beaker. This solution was placed on a hot plate and stirred for several hours to melt and homogenize the IPP resin with soybean oil. After the formation of solution, dried WKF with approximate diameter of 8 cm was dipped in the prepared solution for about 10s. This was followed by quenching at different temperatures at 20, 30 and 40°C in ultrapure water and kept it for several hours. The soybean oil from the membranes was extracted using n-hexane. On complete oil extraction, the membrane color turns yellow to pure white. The prepared membranes were then dried in an oven at 80°C for 8 hrs to remove moisture. This process has resulted in the formation of the homogeneous white colored IPP film on the both sides of the WK fabrics. (Fig. 12) elucidates the complete schematic of the abovementioned fabrication route.

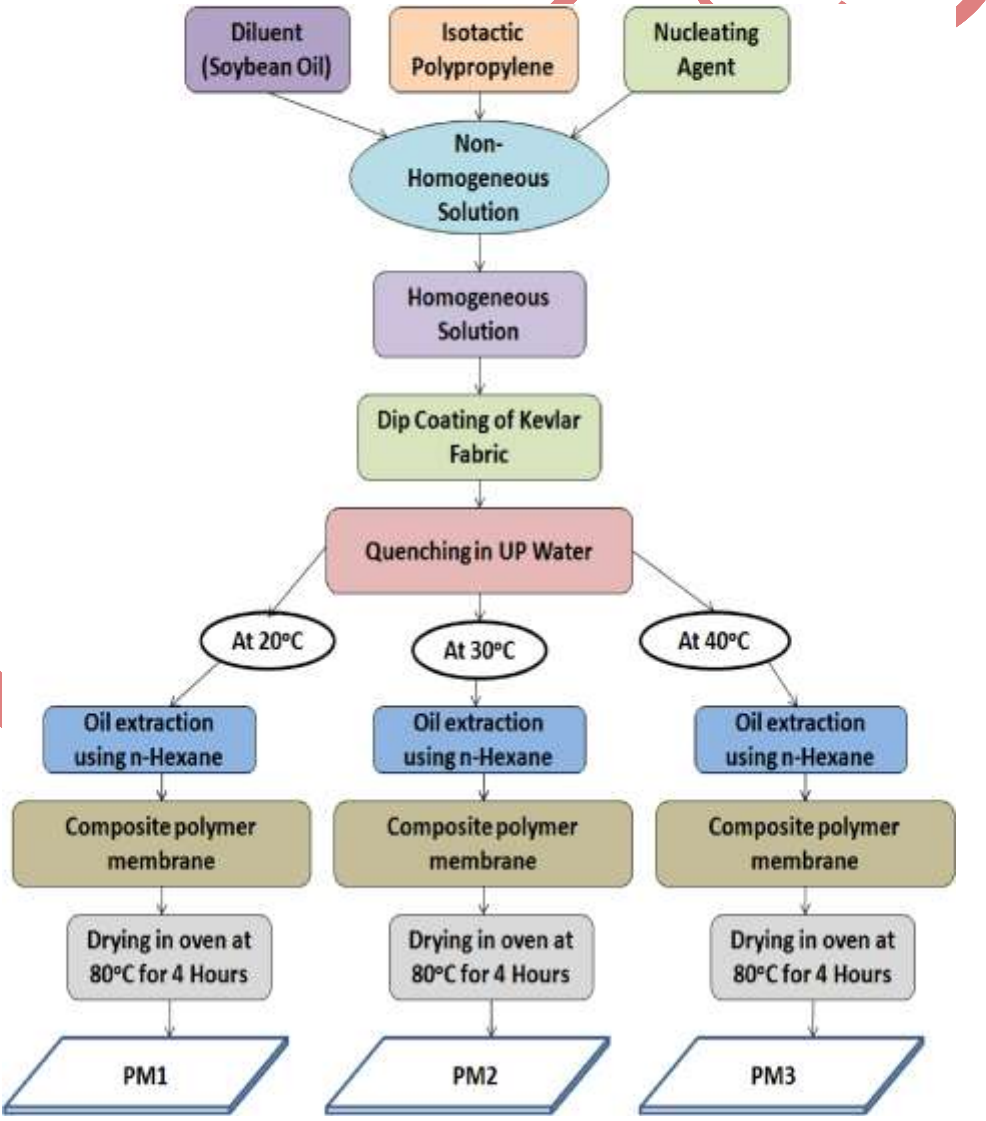

Figure 12. Schematic illustration of the IPP micro-porous membrane preparation at variant quenching temperatures

IV. CHARACTERIZATION TECHNIQUES

The surface morphology and developed microstructure of the fabricated membranes were analyzed using scanning electron microscopy (SEM), Jeol JSM 6490A and atomic force microscopy (AFM) Joel JSPM-5200. For SEM analysis of the membranes, gold sputtering machine was used to perform gold coating on the surface of the composite specimen to make them conductive for microscopic analysis. Membrane samples were cut into 0.25 cm^2 pieces. The membrane pieces were mounted on the metal plates with carbon paste. In addition to SEM analyses, membrane surfaces were also analyzed using AFM by operating it in the AC or tapping mode (TM).

4.1 Micro-filtration cell

A domestically manufactured stainless steel nano-filtration cell was used to test the flux characteristics of variant solvents through the fabricated membranes. Schematic of the process is shown below (Fig.13). The performance of the prepared membranes was experienced at different nitrogen pressures for the flux behavior of different solvents such as water, methanol, ethanol and iso-propanol[8, 15]. For this motive, nitrogen gas was supplied from a nitrogen gas cylinder to nano-filtration membrane cell to attain desirable pressure to permeate the liquid through the prepared porous membranes.

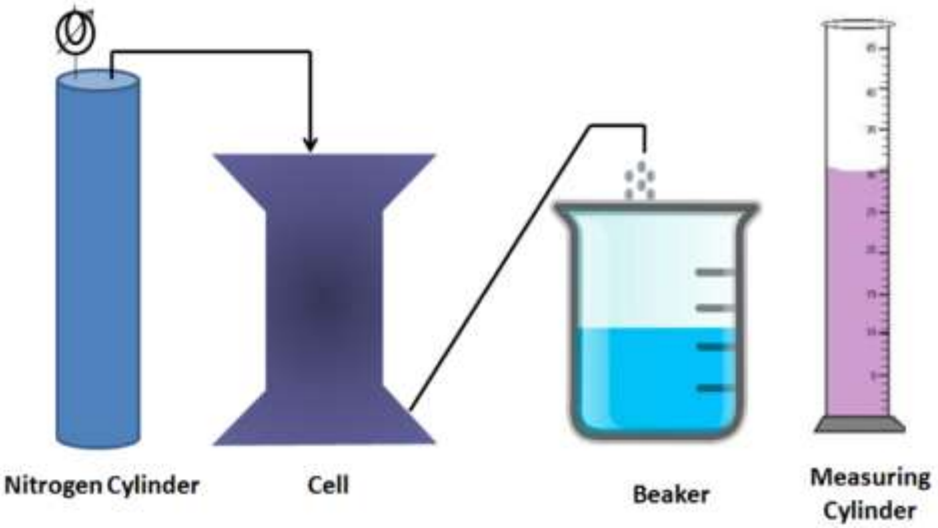

Figure 13. Schematic of the filtration process employed to test the performance of the fabricated IPP membranes.

For each experiment, 500 ml of liquid was taken in the cell and then subjected to the required pressure.The fabricated samples of the membranes were employed to the permeation experimentations for pure solvents at variable pressure of N_2 gas i.e. 13, 26, 39 psi to measure the feed flux through the fabricated membranes. The solvent permeate was collected in a cylindrical jar to measure its volume. The permeability (P_{SP}) was calculated in $m^{-2}\ bar^{-1}\ h^{-1}$ units using Equation 1

$$P_{SP} = Q/PA \qquad (1)$$

Where Q is the volumetric flow rate (l/h) for the solvent permeation, A is the effective filtration area (m^2), and P is the trans-membrane pressure drop (bar).

The pure water and other solvents flux, Q [$L/(m^2h)$] of the membrane was determined by direct measurements of permeate volume, which was calculated by the following Equation 2

$$Q = V/At \qquad (2)$$

Where V was the volume of permeated solvent; A is the effective membrane area; and t is the permeation time[26, 34]

V. CONCLUSIONS

Micro-porous IPP membranes were prepared on weave Kevlar fabric by TIPS techniques. During the fabrication, all parameters were kept constant except the variation of the quenching temperature. The structure of the fabricated membranes was investigated using SEM and AFM. The permeation of various solvents: water, methanol, ethanol and iso-propanol were tested using a custom made filtration cell. It is observed that with the increase in quenching temperatures, for the fabricated membranes, porosity has increased. Average Pore sizes are observed to be 0.925 µ, 2.850 µ and 3.250 µ, respectively for 20, 30 and 40°C quenching temperatures. The flux characteristics of the prepared membranes were also tested for the prepared membranes at various pressures for different solvents in the filtration cell. The flux for the membranes prepared at higher quenching temperature found to be highest and thus support the mciro-porosity information obtained from SEM analysis. Furthermore, flux rate of water observed to be highest while iso-propanol was relatively lower for the membranes prepared at higher quenching temperature. This observation was explained in consideration of the smaller size of water molecules relative to other solvent molecules tested in the current work. It is also observe that with the increase in pressure, flux has also increased.

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