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KEVLAR SUPPORTED NOVEL POLYPROPYLENE MEMBRANES: EFFECT OF POLYMER CONCENTRATIONS ON THE PROPERTIES AND PERFORMANCE

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ABSTRACT

Novel membranes were fabricated using weave Kevlar fabric sandwiched between two isotactic polypropylene (IPP) layers and employing varying concentrations of IPP while keeping constant concentrations of adipic acid as the nucleating agent as well as other conditions. 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. It is observed that with the increase in the concentrations of the IPP in the fabricated membranes, porosity and pore sizes found to reduce. Average Pore sizes are observed to be 2.8, 2.54, and 1.718 µm, respectively for 10, 15 and 20 wt% concentrations of IPP. 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 results obtained indicate the dependence of flux on three factors: types of solvent, applied pressure and fabricated membrane. The flux for the solvents observed to diminish with the increase in concentrations of the IPP in the prepared membranes and is attributed to the decrease in the pore density and pore size.

I. INTRODUCTION

Various types of Synthetic membranes are extensively used in many commercially and technically relevant processes including purification of bio-products and food stuff, sea and brackish water desalination, and the separation of gases and vapors[1]. They are also key components in energy conversion and storage systems, in artificial organs and drug delivery devices[2].There are many techniques used to prepare the synthetic membrane including polymer, ceramic, and metallic membranes. Polymer membranes are more abundantly used in the purification and separation processes due to their excellent tunable characteristics according to the requirement [3]. The polymer membranes used in microfiltration, ultra-filtration, reverse osmosis, and gas separation processes are developed using thermally induced evaporation process (TIPS), thermally assisted evaporation process (TAEP), controlled thermal stretching of polymeric films, and polymer grafting[4, 5] The TIPS is mainly used to synthesize micro-porous membranes ranging from 0.1 to 10µm pore size distribution.

The TIPS is pertinent to the polymers that could not be used in the conventional phase inversion membrane formation due to the solubility problems. The membranes fabricated through TIPS have goo antifouling properties. The simplestmethod for obtaining a porous membrane is by cooling down a two component polymer- solvent mixturein which at sufficiently high temperature forms a homogeneous solution, but at a lower temperature elucidate a miscibility gap over a wide range of compositions[6, 7]. If the polymer is highly crystalline further liquid-solid de-mixing can take place resulting in a pure crystalline polymer in equilibrium with a liquid polymer solution[8, 9]. If the polymer concentration in the polymer rich phase has reached a certain value, its viscosity is increased to such an extent that it can be considered as solid. Or if the polymer is highly crystalline the polymer rich phase will further change into pure crystallized polymer and a polymer saturated solution [10, 11]. The polymer rich phase forms the solid membrane structure and the polymer lean phase the liquid filled pores. A supported liquid membrane (SLM) is one of the three phase liquid membrane systems in which the membrane phase is detained by capillary forces in the pores of micro-porous polymeric or inorganic film [12]. The immobilized liquid is a membrane phase and a micro-porous film or fabric serves as a support for the membrane. Typically, SLMs are used in liquid-liquid and solid-liquid separation[13]. The unique flexibility and ease of preparation of SLMs in various configurations, despite some stability and lifetime problems, has resulted in their application in many, sometimes very different fields where selective and efficient separation methods are necessary: in hydrometallurgy, biotechnology, wastewater treatment, the capture of greenhouse gases, analytical and environmental chemistry, and in the pharmaceutical industry[14, 15, 16]. In the present study, novel Kevlar supported isostatic polypropylene membranes are fabricated using TIPS coupled with dip coating technique. The effect of variant polymer concentrations on the pore size, voids distribution, and flux of variant solvents through the prepared membranes is investigated in detail.

II. RESULTS AND DISCUSSION

2.1 SEM Analysis

SEM was used to study the surface morphology of the prepared membranes. SEM images of the Kevlar fabric used were also obtained and presented in (Fig1).

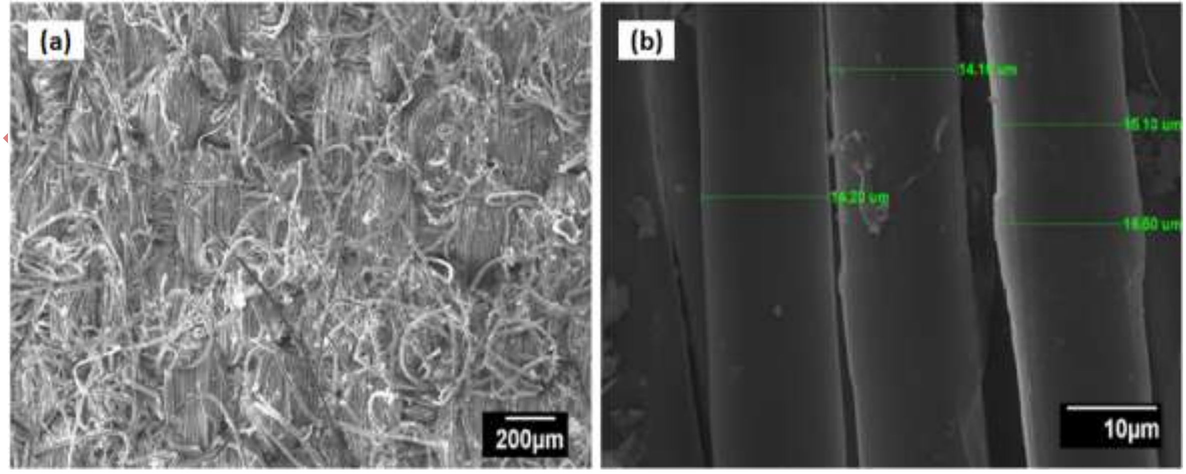

Figure 1. SEM images of weave Kevlar fabric employed for the fabrication of membranes at different magnifications.

(Fig.2-4) represent and compare the surface morphology and extent of pore generation for the three different membranes synthesized using three different polymer concentrations 10, 15, 20 wt% were obtained at various magnifications.

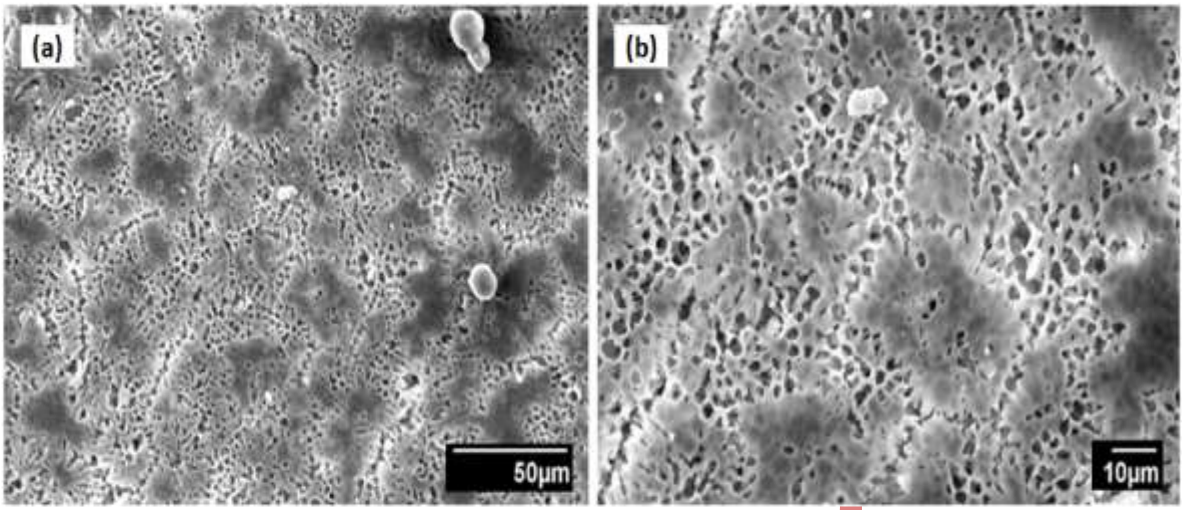

Figure 2. SEM images of the membrane prepared with 10 wt% concentration of IPP at different magnifications

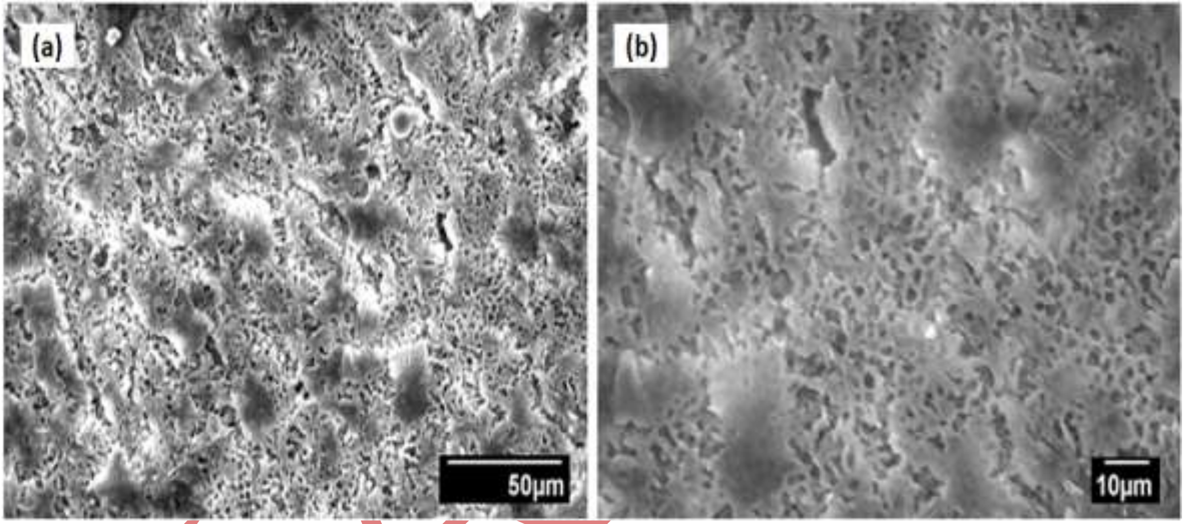

Figure 3 SEM images of the membrane prepared with 15 wt% concentration of IPP at different magnifications

Scanning electron microscopy images showed that membrane porosity increased with decreasing IPP concentrations in the casting solution. It is possible that the growth rate of crystalline IPP increased with increasing polymer concentration during the quench process, so the porosity of the resultant membranes decreased [17]. The determination of the pore size distributions in the prepared membranes indicates the average pore size around 1-2 micron. Since the membrane concentration distribution was narrow for flat sheet IPP membranes preparation, therefore, the influence of IPP concentration on the pore size distribution of the resultant membranes was small [1, 4, 18]. The decrease in pore size with increasing polymer content from 10-20 wt% is due to two factors. Firstly, as the polymer concentration increases, there is less time for coarsening of droplets due to the shorter time interval while going from the binodal to crystallization temperature (T_c) [17]. Secondly, coarsening of the droplets is slower for higher polymer concentrations due to the higher viscosity of the polymer-rich matrix phase and the smaller polymer-lean droplet phase volume fraction [19-21].

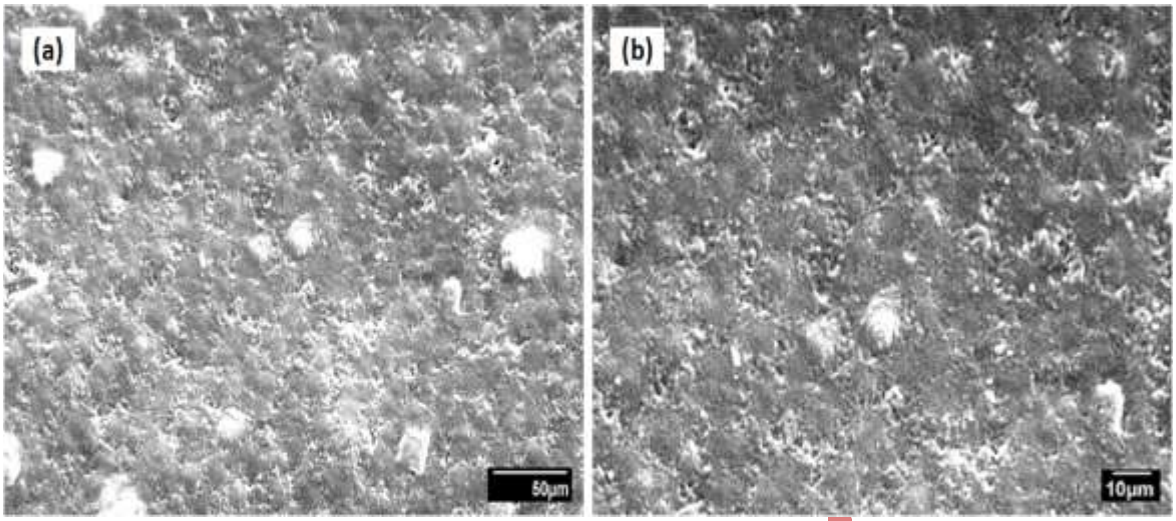

Figure 4 SEM images of membrane prepared with 20 wt% concentration of IPP at different magnifications

(Fig. 5) elucidates that the porosity in the 20 wt% sample is smaller than in the 15 and 20 wt% samples. This can be explained by the smaller volume fraction of polymer-lean phase in the 20 wt% sample. Despite the fact that the 20wt% sample underwent solid±liquid TIPS prior to droplet formation, no spherulites were detected.

2.2 AFM Analysis

Representative AFM images of the prepared IPP-membranes with different concentrations of IPP are shown in (Fig.6-8). The results of the AFM imaging illustrate 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[22, 23]

2.3 Flux Rate Measurements

The performance and efficiency of the prepared membranes were investigated by the measurements of the flux rate. For this purpose, four different liquids were used, which possess different properties. These liquids were:

- Water
- Methanol
- Ethanol
- Iso-Propaonol

Tabel 1. Summary of the properties of liquids employed to test the performance of the prepared IPP-membranes

Property CAS No.	Water 7732-18-5	Methanol 67-56-1	Ethanol 64-17-5	Iso-propanol 67-63-0
Molar mass (g/mol)	18.01528	32.04	46.07	60.1
Molecule Size (nm)	0.26nm	0.41 nm	0.44nm	0.48nm
Density g/cm3 (20 °C)	1.0	0.7918	0.789	0.786
Dipole moment (D)	1.85	1.69	1.69	1.66
Viscosity Pa s (20 °C)	0.001	0.00059	0.0012	0.00286

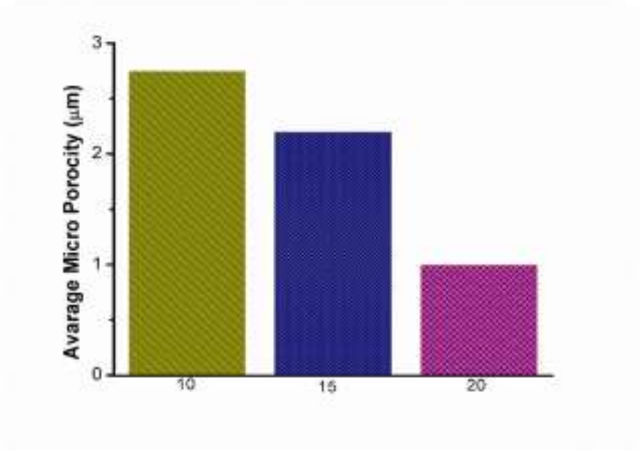

Figure 5.Effect of IPP concentration on the average porosity of the fabricated membranes

In accordance to the data given in Table 1, 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 the 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[18, 21, 24] 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, 26 and 39 psi were applied and graphs between pressure and the flux rate were recorded.

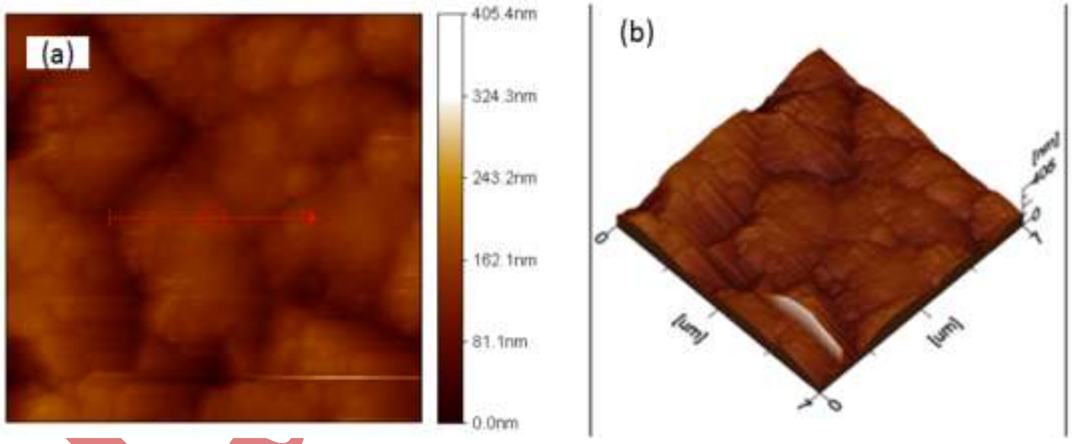

Figure 6.AFM topographical images of the membrane prepared with IPP concentration of 10 wt%

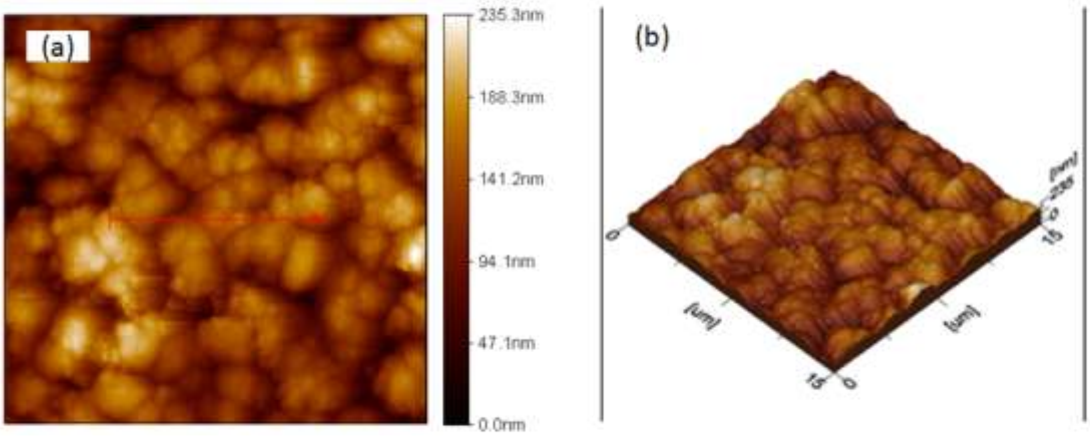

Figure 7.AFM topographical images of the membrane prepared with IPP concentration of 15 wt%

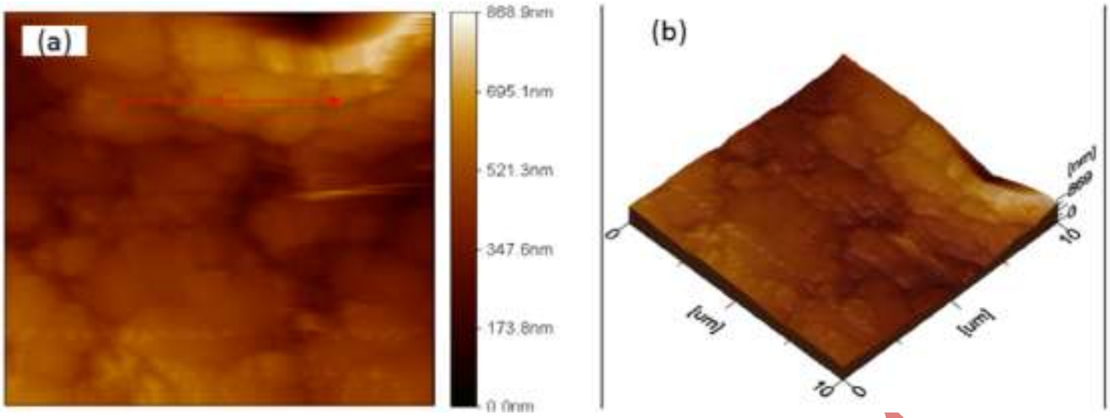

Figure 8. AFM topographical images of the membrane prepared with IPP concentration of 20 wt%

(Fig.9-12) represents results of the flux rate of different solvents tested for the IPP-membranes prepared at various IPP concentrations of 10, 15 and 20 wt%, respectively.

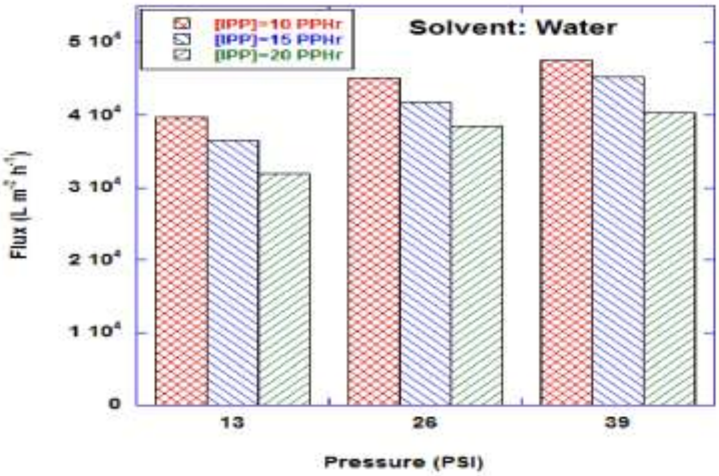

Figure 9.Effect of IPP concentration on the flux behavior of Water through the fabricated membranes at variant gas pressures

The results obtained simulate the dependence of flux on the type of solvent, pressure as well as the membrane. It is observed that with the increase in pressure, the flux rate of the solvents is also increased.

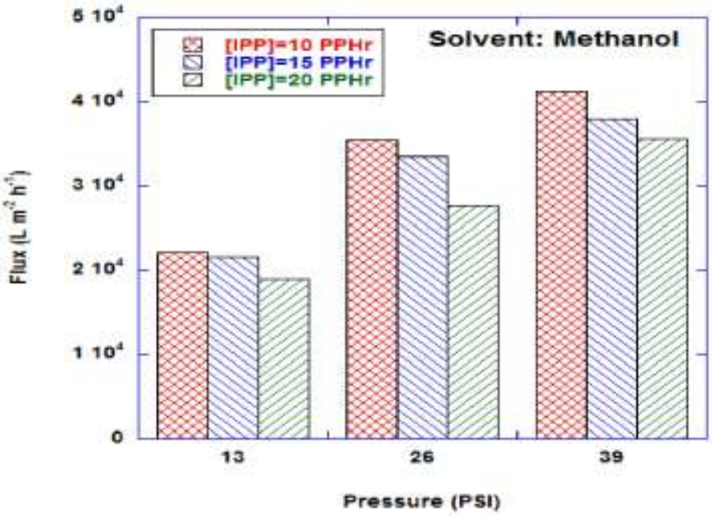

Figure 10.Effect of IPP concentration on the flux behavior of Methanol through the fabricated membranes at variant gas pressures

Furthermore, it is noted that in general water has the maximum flux and isopropanol has the minimum value. This observation can be correlated to the size of the liquid droplets employed in the present work.

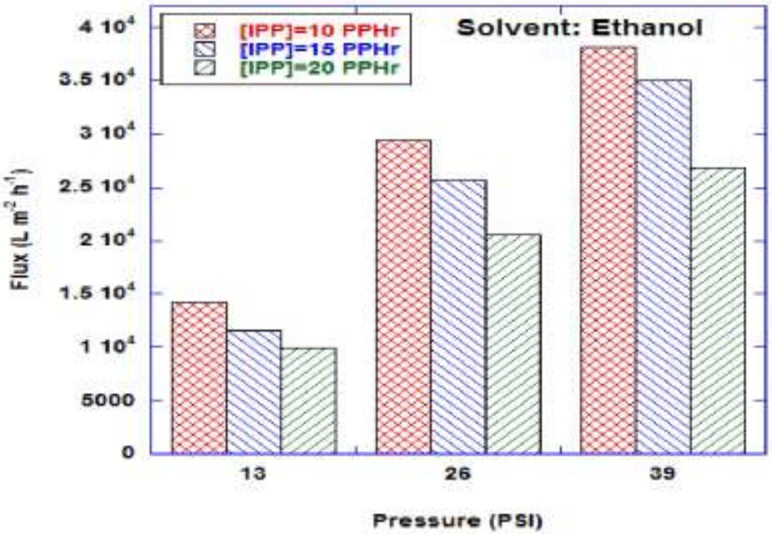

Figure 11. Effect of IPP concentration on the flux behavior of ethanol through the fabricated membranes at variant gas pressures

It is interesting to observe that the flux for the solvent increase with the decrease in the IPP concentration in the prepared membranes and this can be attributed to the decrease in the porosity size distributionwithin the prepared IPP-membranes.

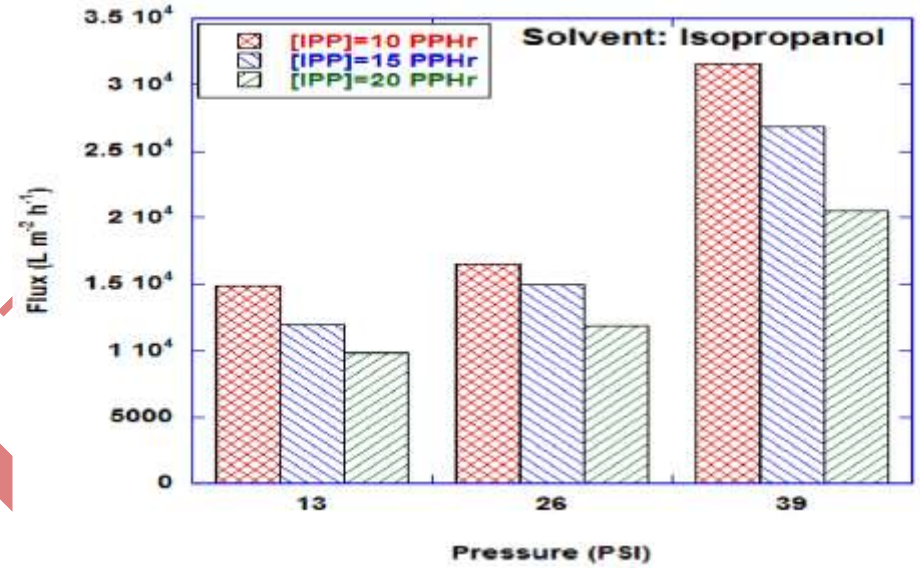

Figure 12.Effect of IPP concentration on the flux behavior of iso-propanol through the fabricated membranes at variant gas pressures

The variable concentrations of IPP may have affected the degree of nucleation and hence crystallization, which can significantly modify the crystallization behavior of IPP. It is possible that IPP crystal nucleation and crystal growth was affected during the phase separation process to influence the formation of crystalline, spherulites dimensions and thus crystallinity [25]. These factors may have resulted in relatively larger pores and also higher density to affect the porosity of the fabricated membranes at lower IPP concentration.[4 ,26]. The observed results are in good agreement with that of SEM for the IPP-membranes investigated in the current work. The flux-pressure curves delineate three observations; 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[27]; 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[27].The third study of this investigation is that with increasing the polymer concentration in the membrane solution, solvent flux through the membrane reduces due to the pore size reduction as observed in SEM images[28].

2.4 TGA Studies

(Fig.13) represent the effect of temperature on the thermal decomposition of the supported fabric and the prepared membranes. Effect of temperature change on both the Kevlar fabric and IPP membrane indicate the temperature at which the prepared membranes can undergo thermal degradation.

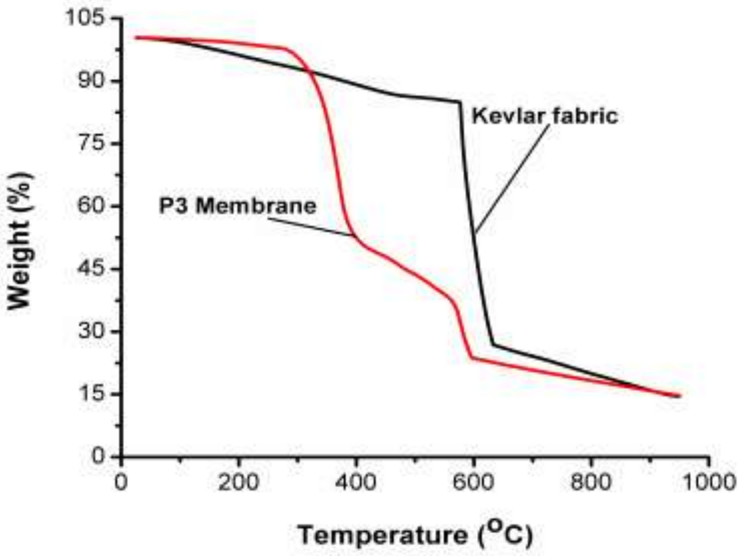

Figure 13. Representative thermal decomposition contours of the Kevlar fabric and the fabricated membrane with 20 wt% polymer concentration

From the TGA of Kevlar, it could be seen that decomposition took place in three unique steps. In the first step plain Kevlar fabric, shows weight loss of around 10% between 20 and 620°C that may be due to the presence of volatile components. In the second step a very sharp decomposition take place at around 620-630 °C with a significant weight loss of about 60%. In the third step, there was another weight loss of about 20% from 630 to 950°C. Both the stages 2 and 3 are probably due to main thermal decomposition and/or due to the carbonization of the decomposed products to ash of the Kevlar chains[29-31].The TGA study of the prepared membranes indicates slightly different behavior. It is evident that the decomposition took place in four unique steps. In the first step, membrane shows slight weight loss of around 5% between 20 and 300 °C that may be again attributed to the presence of volatile components. In the second step, a very sharp decomposition take place at around 300-400°C with a significant weight loss of about 40%. In the third step, there was another weight loss of about 25% from 400 to 600°C. In the final stage of step 4, another 10% thermal decomposition was observed in between 600 to 950°C. As in the case of Kevlar, the thermal analysis for the membranes suggests the main thermal decomposition and/or due to the carbonization of the decomposed products to ash of the polymer chains[31, 32].

III. EXPERIMENTAL

3.1 Materials

The reagents employed to fabricate membrane were used as received. The membranes were fabricated from isotactic polypropylene (IPP) was purchased beijing Yan De Chemical Industries Co., Ltd, China. Adipic acid and n-hexane were supplied by Sigma-Aldrich. Pure soybean oil was purchased from weifang Olin Import and Export Co., Ltd. To support the membrane, weaved Kevlar fabric (WKF) was supplied by DuPont International, USA.

3.2 Fabrication of Membranes

The membranes were fabricated using thermally induced phase separation (TIPS) procedure. One main difference in this work was the employment of the variable IPP concentrations to prepare SLMs membranes. Typically, in the first step, suitable amount of soybean oil was taken in a 500 ml beaker and then added varying amount of IPP. Three concentrations of IPP with 10, 15 and 20 wt% were used to fabricate SLMs with variant polymer loadings. After this, 0.5wt% nucleating agent (adipic acid) was inserted in the solution. This solution was placed on a hot plate at 220°C and vigorously stirred for several hours to melt and homogenize the IPP resin with soybean oil. After the formation of uniform solution, dried WKF with approximate diameter of 8 cm was dipped in the prepared solution for 10s. This was followed by quenching it in ultrapure water at 30°C. The soybean oil from the membrane was extracted by placing the SLM in n-hexane for 12 hrs. On complete oil extraction from the membrane, its color turns from yellow to pure white. The prepared membranes were then dried in an oven at 80°C for further 6 hrs. This process has resulted in the formation of the homogeneous IPP film on both sides of the WK fabrics.

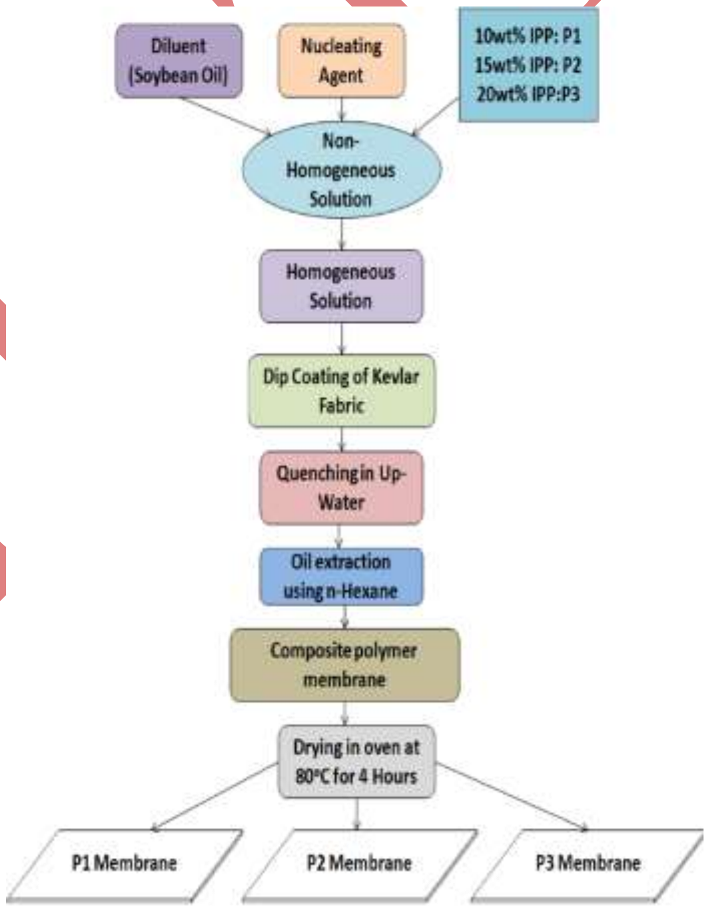

Figure 14.Fabrication schematic illustration of Kevlar supported IPP membranes

IV. CHARACTERIZATION OF MEMBRANES

The characteristics and performance of the prepared membranes were investigated by various techniques.

4.1 SEM and AFM Analyses

Morphology and the microstructure of the prepared membranes were studied with the aid of scanning electron microscopy (SEM), Jeol JSM 6490A and atomic force microscopy (AFM) Joel JSPM-5200. For SEM analyses, membranes were sputtered with gold coating in a gold sputtering assembly. Scanning electron microscopy (SEM) was performed on the Kevlar supported IPP membranes. Membrane samples were cut into 0.25 cm^2 (0.5 cm×0.5 cm) pieces. The membrane pieces were mounted on metal plates with carbon paste and gold-coated prior to use. In addition to SEM analyses, membrane surfaces were also investigated using AFM by operating it in the AC or tapping mode (TM).

4.2 Flux Experiments

A custom made stainless steel cell was used to test the permeation characteristics of the fabricated membranes. Schematic illustration of the experimental flux study through the micro-porous membranes is displayed in Fig 2.

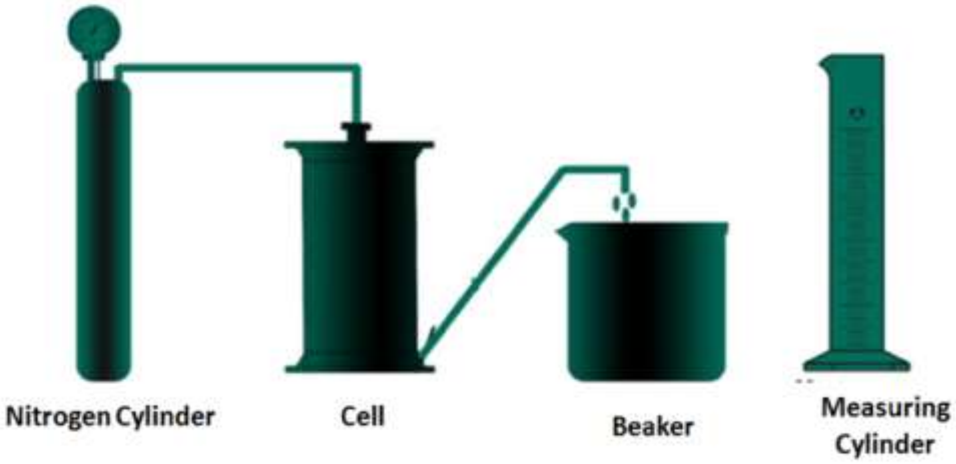

Figure 15.Schematic diagram of the filtration process employed to test the flux performance of variant solvents through the fabricated IPP membranes

The performance of the prepared membranes was tested at different pressures for the permeation behavior of different solvents such as water, methanol, ethanol and isopropanol. For this purpose, filtration membrane cell was attached to a nitrogen gas cylinder to obtain desirable pressure to permeate the liquid through the prepared porous membranes. For each experiment, 500 ml of liquid was taken in the cell and then subjected to the desirable pressure.

The fabricated samples of the membranes were subjected to the permeation experimentation for pure solvents at variable pressure of N_2 gas i.e.13, 26, and 39 psi to measure the permeability. 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.

4.3 TGA Studies

Thermo gravimetric Analysis (TGA)/differential thermal analysis (DTA) studies of the prepared membranes were carried out using a Perkin Elmer Diamond TG/DTA, at a heating rate of 10 °C/min under oxygen atmosphere. Thermal decompositionbehavior of polymer composite membranes was determined from the corresponding curves.

V. CONCLUSION

Microporous IPP membranes were prepared on weave Kevlar fabric by TIPS techniques. During the fabrication, all parameters were kept constant except the variation of the IPP concentrations. The permeation of various solvents: water, methanol, ethanol and iso-propanol were tested using a custom made filtration cell.It is observed that with increasing IPP concentrations, for the fabricated membranes, pore size has decreased.Average Pore sizes are observed to be 2.75 µm, 2.2 µm and 1 µm, respectively for 10, 15 and 20 wt% concentrations of IPP. 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 polymer concentration found to be the lowest 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. 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, solvent flux has also enhanced.

VI. ACKNOWLEDGEMENTS

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